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DATA ACQUISITION AND COMPUTER CONTROL OF GAS CHROMATOGRAPHS

BY

© PETER COXHEAD

A THESIS

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance a thesis entitled DATA ACQUISITION AND COMPUTER CONTROL OF GAS CHROMATOGRAPHS submitted by Peter Coxhead, B.Sc., in partial fulfilment of the requirements for the degree of Master of Science in Chemical Engineering.

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1. INTRODUCTION

Today, the chemical industry is using more and more analytical instruments to improve control of manufacturing operations and to aid in increasingly complex research projects. A survey (2) in the U.S.A. of over 7000 industrial laboratories showed about 60% use chromatographs; 50% use ultra violet and infra red spectrometers; and 20% use mass spectrometers and x-ray techniques. The use of these sophisticated analytical instruments has created problems for the analytical chemist, which centre around the requirements for rapid processing of the vast amount of data generated by these instruments. Since the analytical laboratory is an essential service function of the chemical plant, the throughput rate and efficiency must be as high as possible. A high laboratory utilization factor is also essential to offset the high cost of equipping, maintaining and staffing these facilities. Consideration will now be given to the use of a Data Acquisition and Control System to monitor and control laboratory instruments, gas chromatographs being taken as a typical example. A literature survey, presented in Appendix D, illustrates the status of some of the industrial computer chromatograph systems and reports the advantages resulting from these installations.

Briefly, gas chromatography (8) is an instrumental technique of separating and analyzing the components of a chemical mixture. Figure 1.1 illustrates the basic components of a chromatograph. The column is continuously eluted with an inert carrier gas - such as helium or hydrogen. The sample of material to be analyzed is injected either manually or automatically, ahead of the column into the carrier gas stream. The column which separates the components of a sample, is generally packed with specially treated solid particles. The detector located downstream

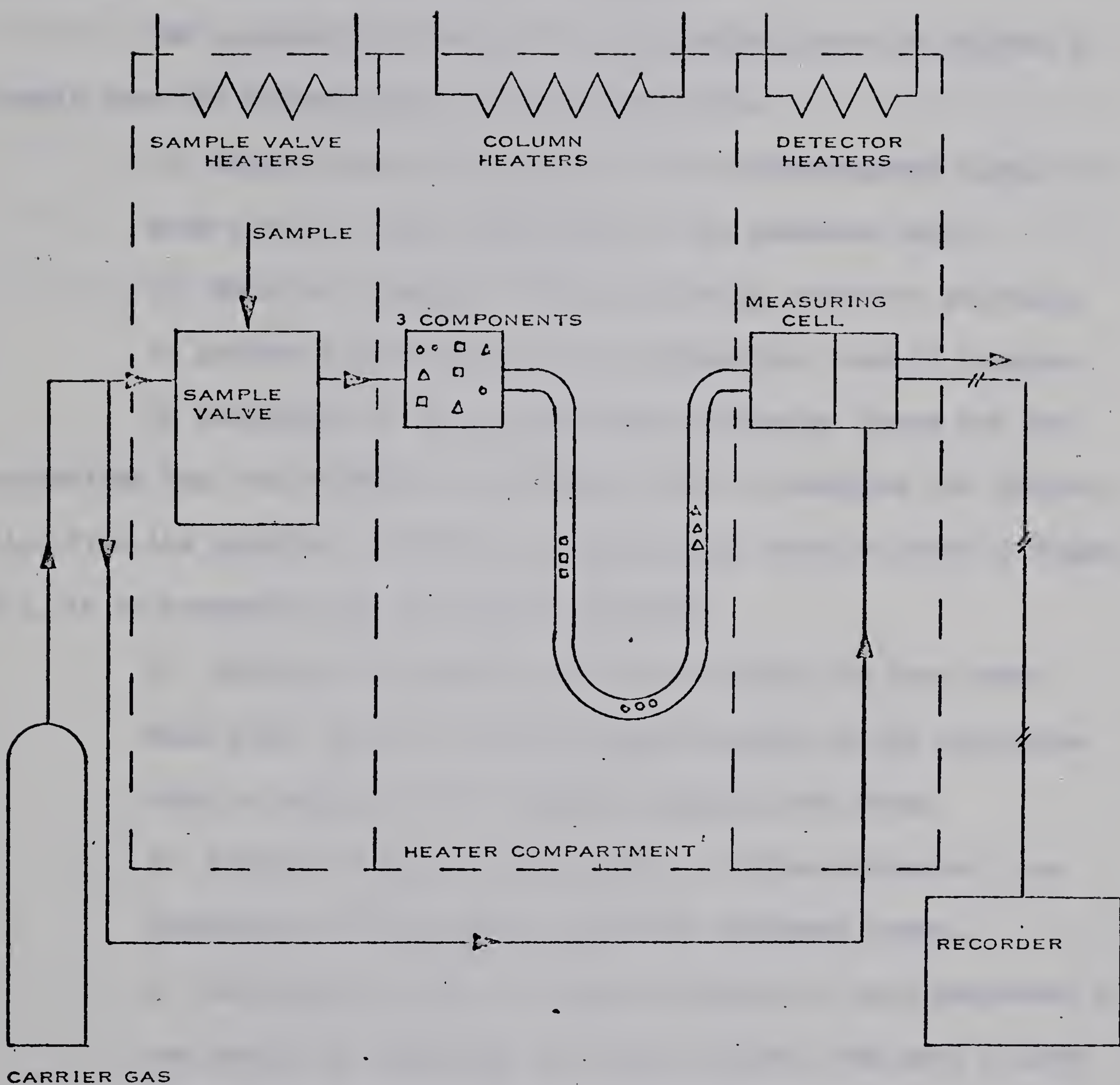


Figure 1.1 Basic Block Diagram of a Chromatograph

of the packed column, senses the arrival of the separated components and produces a signal proportional to the concentration or amount of the particular components in the carrier gas. A perfect separation occurs when each component of the sample leaves the column at different time intervals. Specifications and functional characteristics of a chromatograph will be considered in Chapter 1.

The procedures undertaken by the analyst after he injects a sample into the chromatograph are outlined below:

1) Adjust output attenuation of the chromatograph signal to keep the full scale peak value on the recorder chart.

2) Make the necessary column switching, detector switching or perform backflushing at the appropriate time if required.

On completion of the chromatograph analysis, there are four operations that the technician must carry out to transform the information from the graphical display or chromatogram, such as shown in Figure 6.1, to an acceptable and meaningful analysis:

1) Allowing for baseline drift, calculate the area under each peak, which is related proportionally to the concentration or amount of the component causing that peak.

2) Relate the peaks by comparison to known retention time information to previously specified component names.

3) Calculate the mole or weight fraction of each component in the sample by comparing the areas obtained from part 1 above with the results from a standardization run.

4) Prepare an analytical report for distribution.

All these operations can take anywhere between ½ to 2 hours of the chromatograph technician's time. So far consideration has been

limited to the processing of data from one chromatograph. However, most laboratories have from five to fifty chromatographs handling a large group of samples. Therefore the techniques associated with handling many chromatographs present very serious operating problems.

Since modern data acquisition and control systems can handle acquisition of large amounts of data, analyze and perform extensive calculations with this data, it is apparent that there are many areas where computer control and data analysis could be used in the analytical laboratory to improve speed, precision and efficiency of analyses (12, 13). Chapter 2 discusses the system design for the application of a computer to monitor gas chromatographs.

2. FUNCTIONAL CHARACTERISTICS AND SPECIFICATIONS OF A PROCESS GAS CHROMATOGRAPH

In order to obtain the specifications for the design of a computer program to monitor gas chromatographs, it is essential to know the functional characteristics and specifications of a typical chromatograph, ie. the range and speed of operations to be handled, typical parameter values, etc. For this reason the hardware and operational features of a typical chromatograph will be reviewed in this chapter.

The main features of a basic laboratory chromatograph system are discussed in the introduction. For more complex separation problems encountered in chromatographic analyses, the following features are often added, as shown in Figure 2.1.

1) Stripping valves.
2) Column backflush valves.
3) Multiple columns in series and parallel.
4) Multiple detectors including conductivity, hydrogen flame ionisation, etc. (14).
5) Column temperature programming.
6) Carrier gas flow programming.

These 'hardware' features are discussed below.

2.1 Sampling Section

Multiple process streams may be analyzed with a single chromatograph by using a stream selector (15). Beckman (1) specify for their Model 320-C Process Chromatograph that up to ten similar streams may be analyzed by using their Automatic Stream Selector with maximum analysis capability of six components. An alternative approach to the above stream selection is to utilize ten sampling valves on the process streams. Chemcell Ltd. process chromatograph design department have found by experience that because

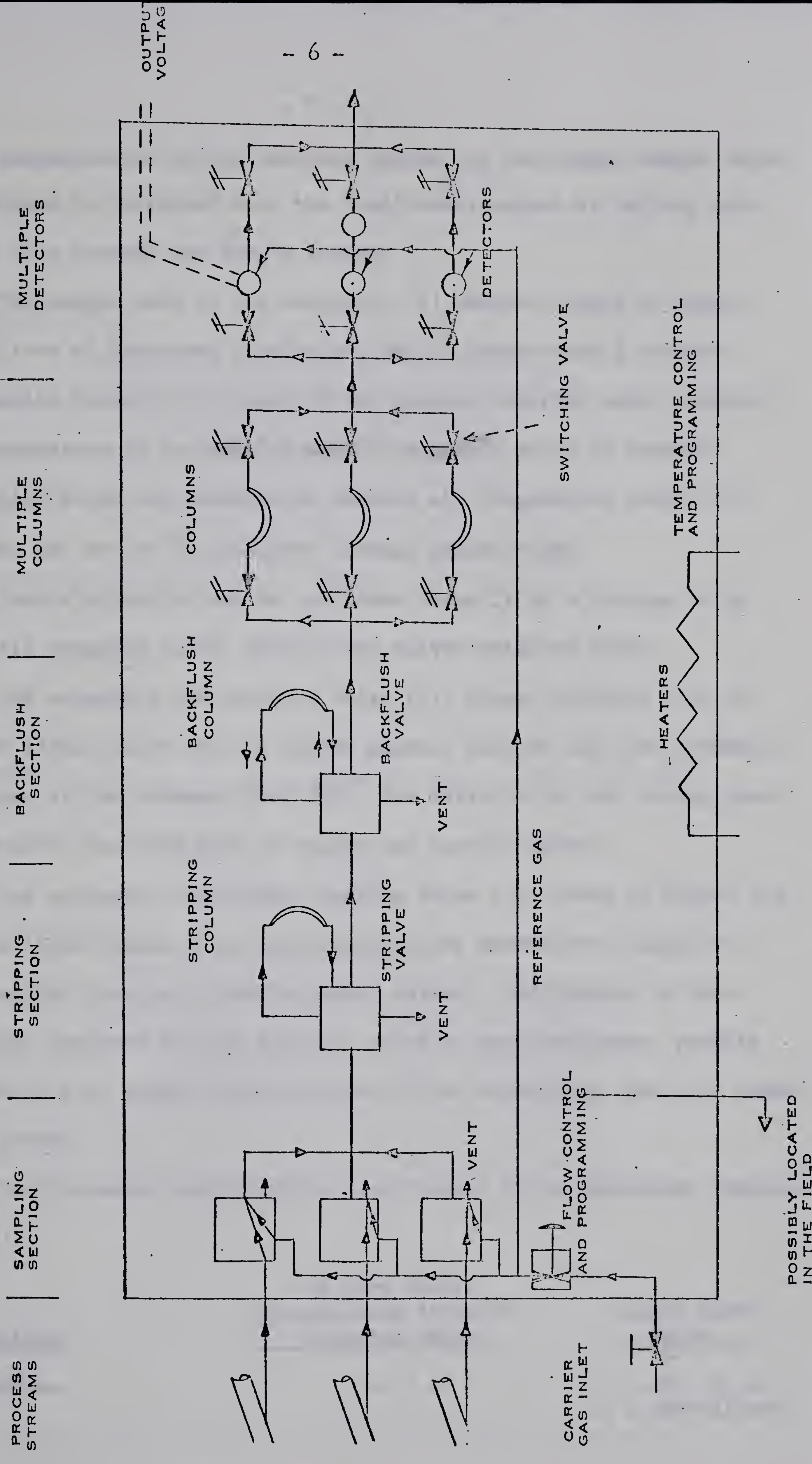
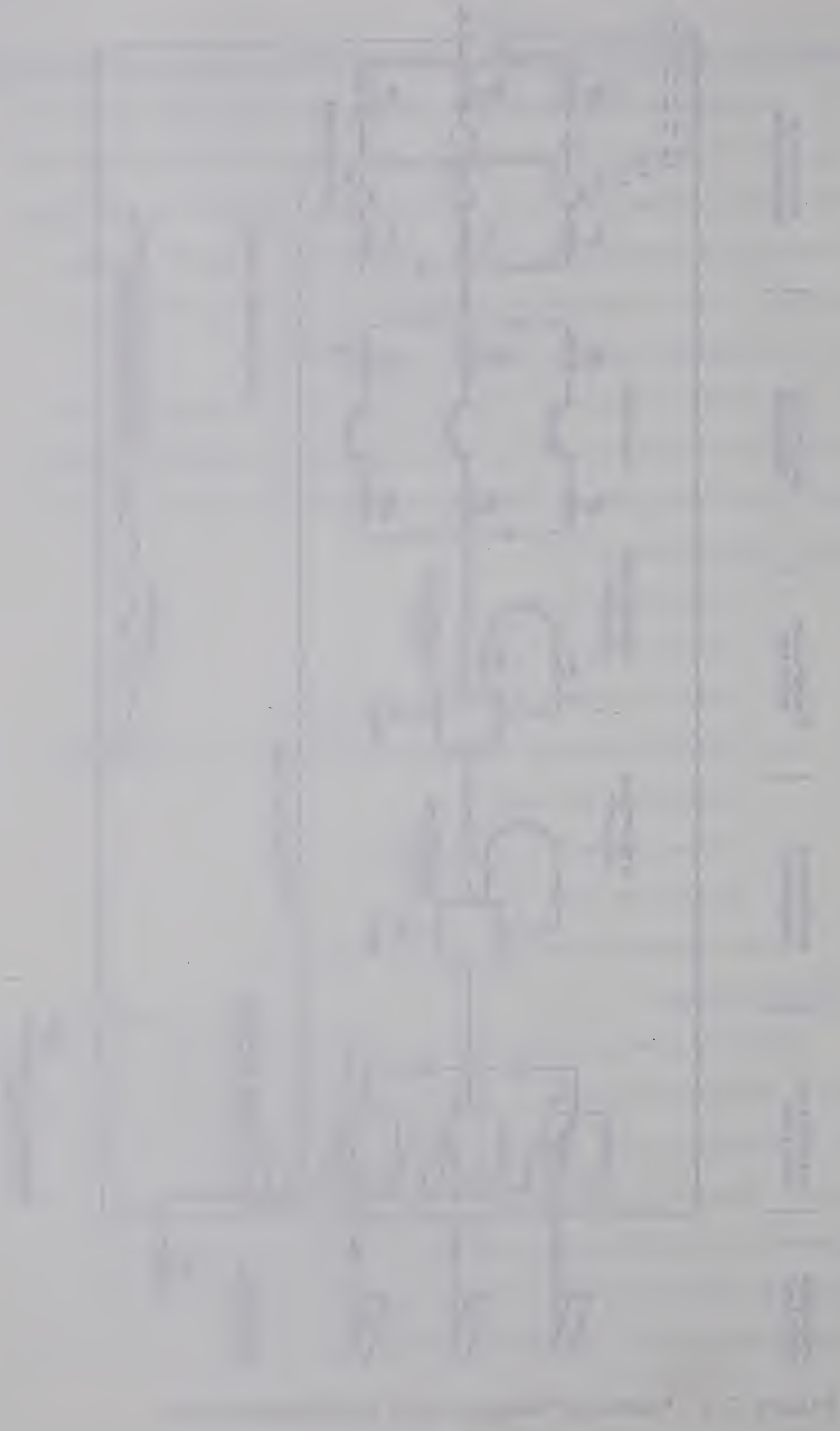


Figure 2.1 'Hardware' Features of a Gas Chromatograph



of cross contamination in the sampling system, an individual sample valve in each stream is preferred over the traditional method of valving each stream in turn through one sample system.

The sample sent to the analyzer, (3) whether liquid or vapor should be free of entrained liquids and solids larger than 5 microns. Liquid samples should be at least 25 psi greater than the vapor pressure at the temperature of the zone in which the sample valve is located. Vapor samples which can condense at ambient air temperature should be passed into and out of the analyzer through heated tubes.

Sample injection may be performed manually by a syringe or by an automatic sampling valve, such as the valves outlined below.

The automatic gas sampling valve (1), shown in Figure 2.2, is a compound linear valve used to inject gaseous samples into the chromatograph column of the Beckman 320-C PGC. The valve is air and spring operated to control the flow path of sample and carrier gases.

The automatic liquid-gas sampling valve (1), shown in Figure 2.3, is used to inject small, from approximately one microliter, liquid or gaseous samples into the chromatographic column. Utilization of this valve, which replaces the gas sampling valve on some analyzers, permits direct analysis of liquid samples without first vaporizing them in a sample handling system.

The following specifications are typical of chromatograph sampling valves (3).

Specifications	16 Port Rotary Pneumatically Actuated Sampling Valve	Liquid Slide Valve
Sample Volumes	1 to 5 cc	.1, .25, .5, 1, 2, 4 microliters

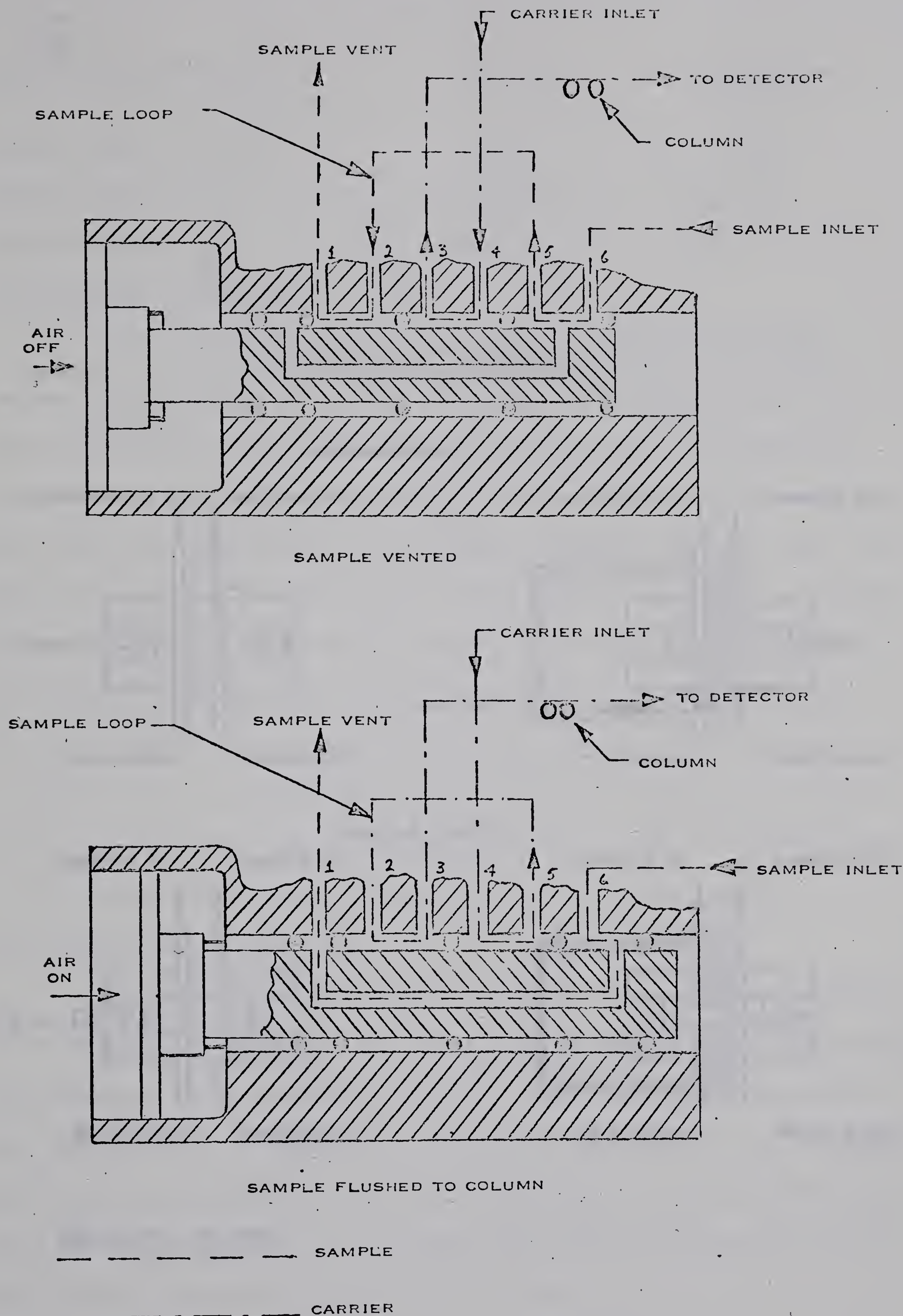


Figure 2.2 Automatic Gas Sampling Valve Flow Diagram

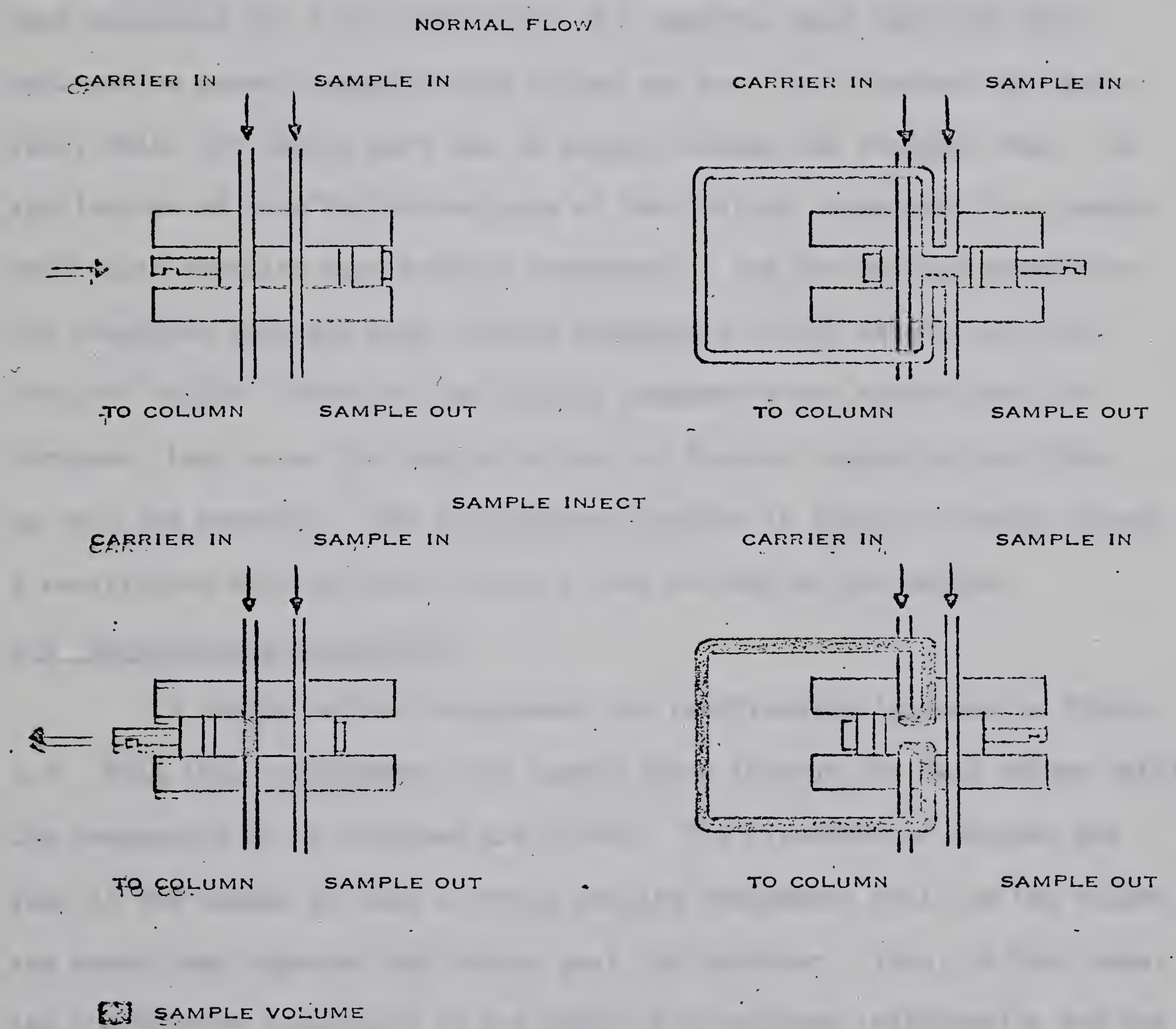


Figure 2.3 Automatic Liquid-Gas Sampling Valve Flow Diagram

Sample Reproducibility	$\pm$ 0.25%	better than $\pm$ 0.1%
Minimum Sample Pressure	1 PSIG	1 PSIG
Maximum Sample Pressure	30 PSIG	150 PSIG
Maximum Temperature	150°C	150°C

2.2 Stripping Section (1)

This section is composed of a stripping valve and stripping column shown in Figure 2.1 and more specifically in Figure 2.4. It is used basically for a pre-separation of a mixture, such that the first part can be passed into the main column and over the detectors for analysis, while the second part can be purged through the stripper vent. An application of this is the analysis of low boiling components in a sample which also contains high boiling components. The low boiling components are separated from the high boiling components in the relatively short stripper column. When the low boiling components are eluted from the stripper, they enter the single column for further separation and then go into the detector. The non-analyzed portion is purged to waste through a restriction with the same pressure drop as that in the column.

2.3 Backflushing Section (1)

A single column arrangement for backflushing is shown in Figure 2.5. With this arrangement, the sample flows through the main column until the components to be analyzed are eluted. The direction of carrier gas flow in the column is then reversed and the components still in the column are moved back together and eluted past the detector. Thus, in this case, the low boiling components of the sample are analyzed individually and the high boiling components are analyzed as a group.

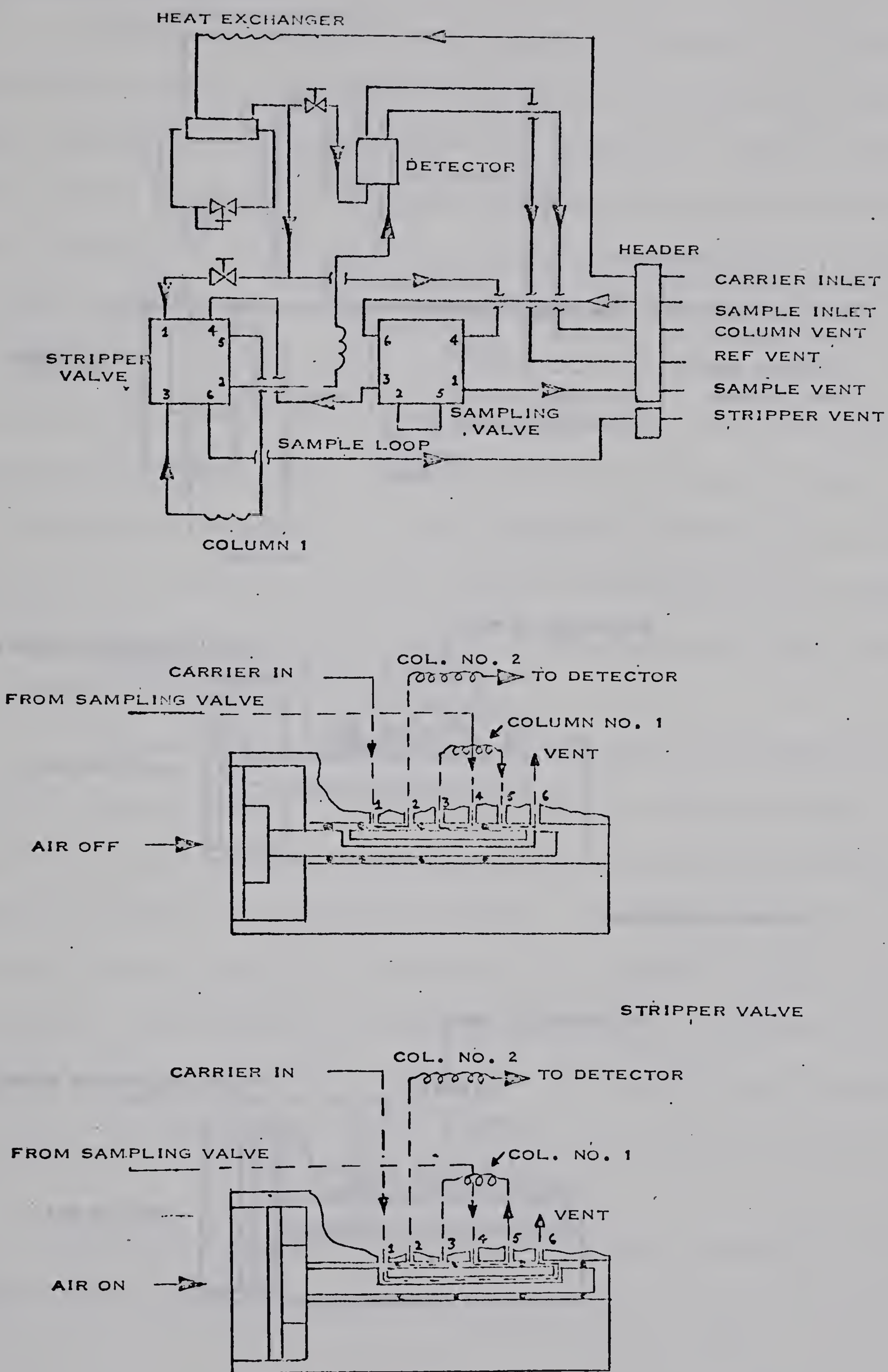
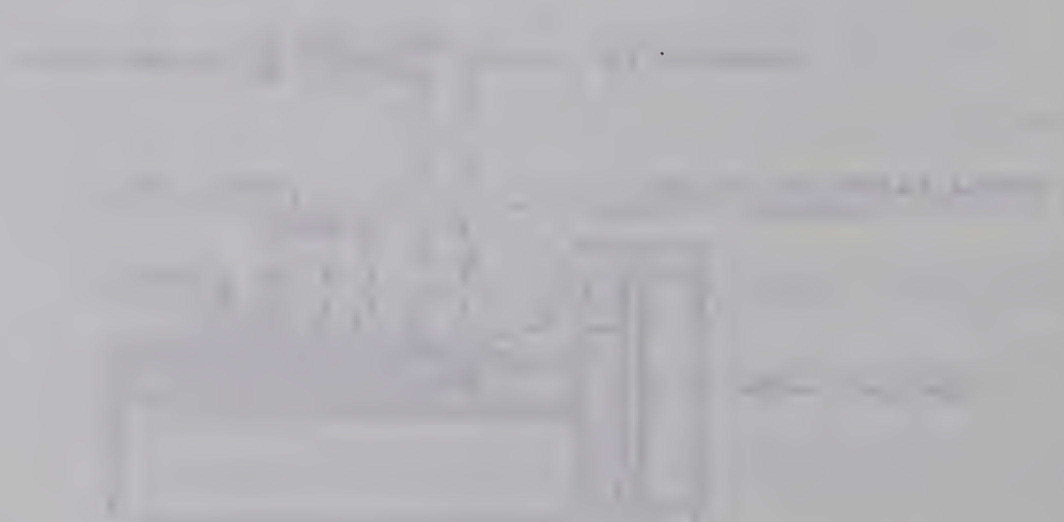


Figure 2.4 Single Column with Stripper Arrangement



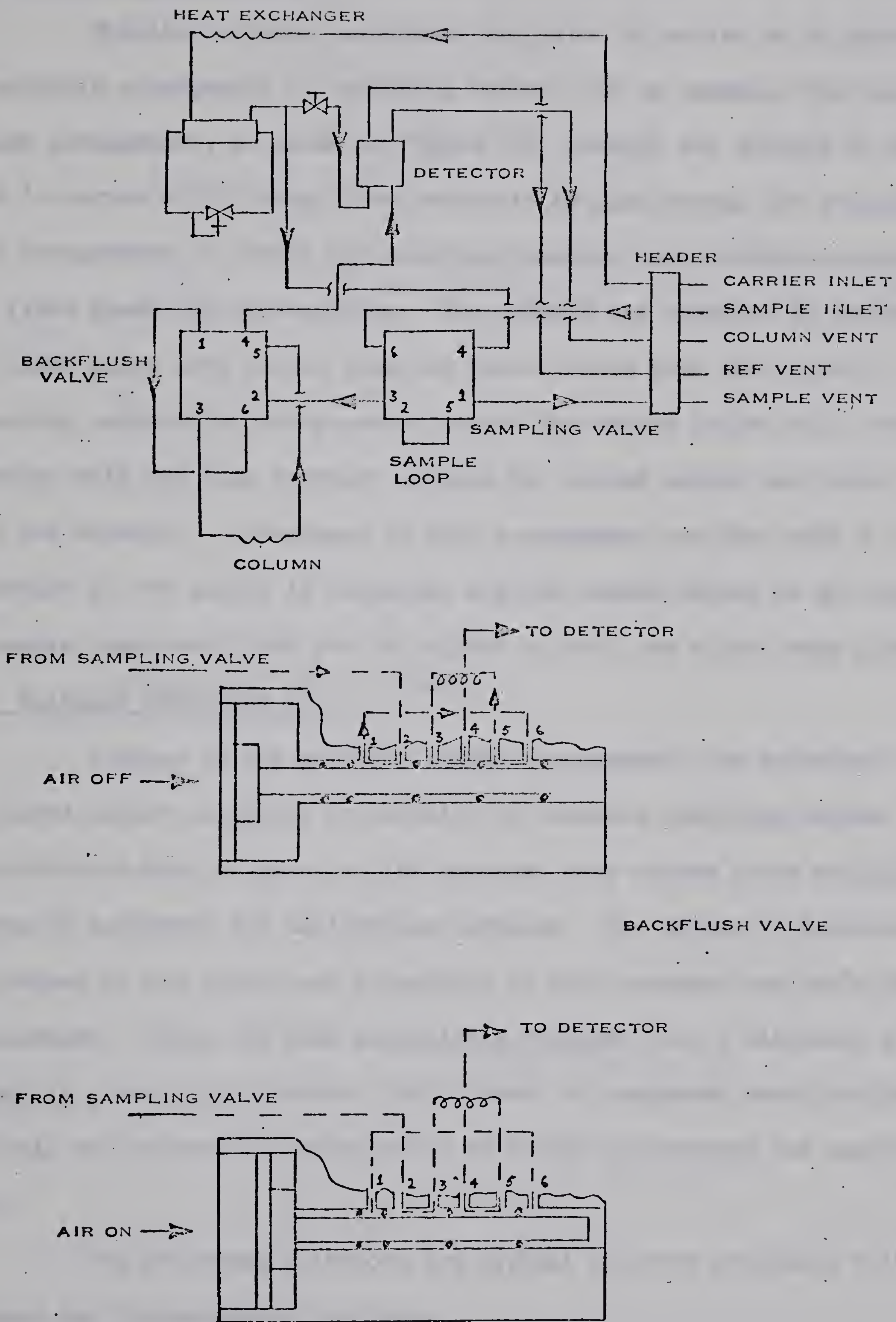


Figure 2.5 Single Column with Backflush Arrangement

2.4 Multiple Columns (1)

Multiple columns can either be placed in series or in parallel by suitable arrangement of switching valves. As an example, the dual column arrangement, as shown in Figure 2.6, permits two columns to be used in series or to change from series to by-pass during the analysis. This arrangement is useful for analyzing samples such as those containing fixed gases and condensibles. The columns are operated in series until the fixed gases have passed from the first column into the second. The remaining components are by-passed around the second column via a restriction with the same pressure drop as the second column and passed over the detector. Advantages of this arrangement are that only a single injection of the sample is required, and the second column is not exposed to sample components that are not eluted or that are eluted very slowly.

2.5 Multiple Detectors (3)

Similar to the multiple column arrangement, the detectors can be placed either in series or parallel by suitable switching valves. Consideration must be given to the pressure drop across these multiple pieces of equipment for calibration purposes. The choice of detector is determined by the amount and properties of the component available for measurement. While the high sensitivity recorder (ie. 1 millivolt full scale) is a desirable feature, the ultimate in component sensitivities can only be achieved by optimization of column performance and sample size.

The following detectors are typical of those available with Process Gas Chromatograph analyzers.

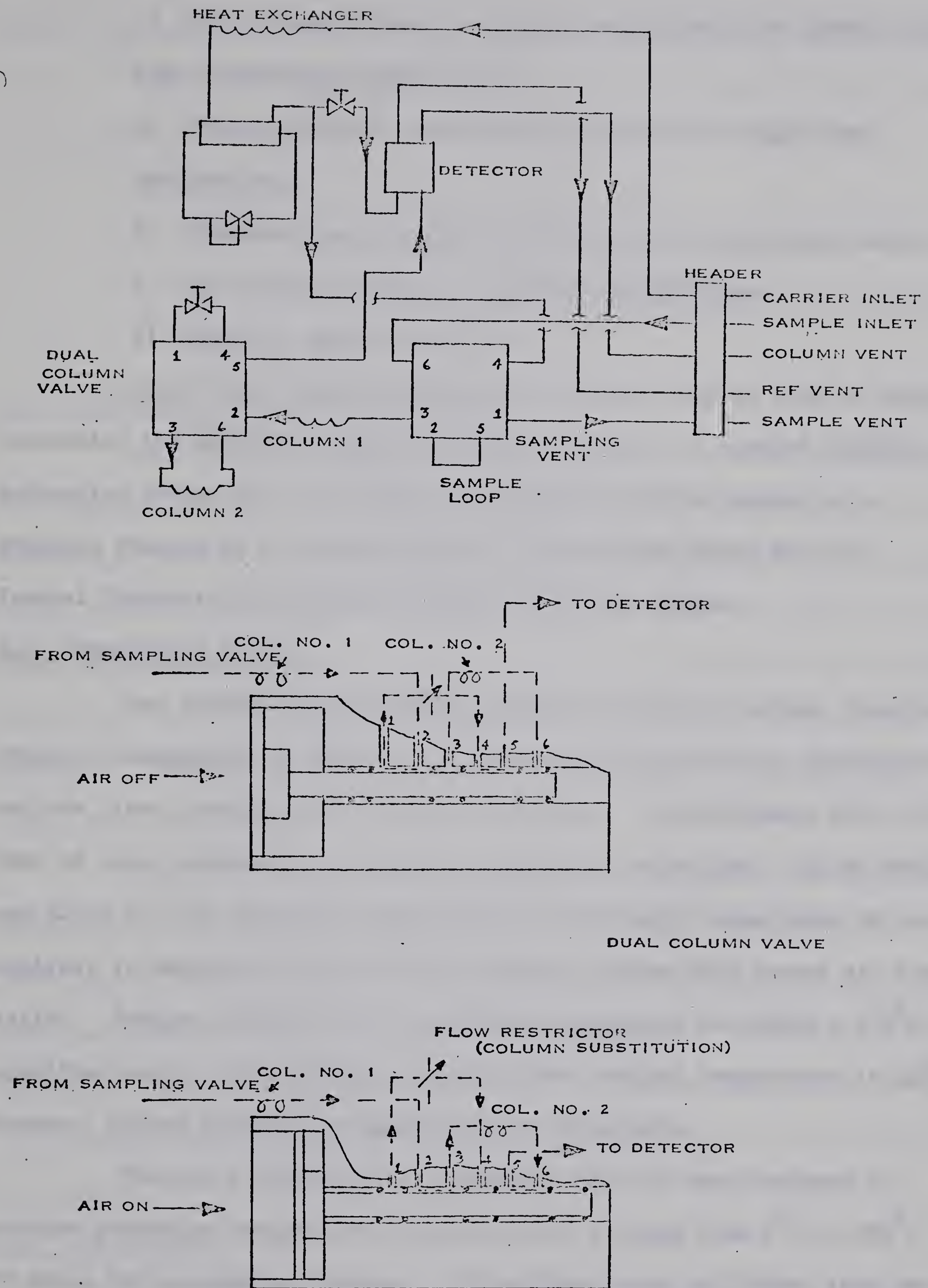


Figure 2.6 Dual Column Arrangement

1) Four element hot-wire thermal conductivity for general and high temperature applications.

2) Micro-thermistor conductivity detector for high speed applications.

3) Hydrogen flame ionisation detector for trace measurements.

4) Gas density balance for special applications.

5) Electron capture detector.

All of the thermal conductivity detectors can be used in series preceeding the hydrogen flame ionization detectors. A current limiting protection device for the thermal conductivity detector should be a standard feature of a chromatograph eg. Perkin-Elmer Model 900 (4) Thermal Conductivity Detector Filament Protection Device.

2.6 Temperature Control

Gas chromatography requires precise control of column, inlet and detector temperature as well as the temperature of the valve compartment and the lines leading from one area to another. In the Beckman GC-4 (5), each of these elements is separately heated and controlled. In the Beckman 320-C (1) the operating temperature of the heated compartment of the analyzer is maintained by an electric heating system with forced air circulation. Precise control of the operating temperature to within $\pm$ 0.1°C is specified over a range between slightly above ambient temperature to 110°C. However, higher operating temperatures are attainable.

The M.S.A. Model 550 (3) Analyzer oven has been designed to provide precision temperature regulation over a range from 0°C to 250°C by using the sub-ambient feature. The analyzer oven is divided into two controlled zones, the outer-hot zone having a temperature range of 50°C to 250°C, the inner-cold zone providing the sub-ambient feature. Air, for

cooling the inner zone, is refrigerated by an external Ranque Hilsch-Vortex tube, selected because of its size, versatility and absence of moving parts.

Over-heat protection to the analyzer, resulting in loss of power to the heaters is provided if:

1) heater skin temperature exceeds 270°C
2) the air flow over the heater drops below 1 C.F.M.
3) the outer zone temperature exceeds 10°C above the controlled temperature.

2.7 Programmed Temperature Control (5)

This feature provides a controlled increase of column temperature during a sample run. General advantages of the temperature programmed chromatograph are that sample components with an extremely wide boiling point range can be analyzed in considerably less time; the shorter retention times of the higher-boiling components produces an increase in peak height sensitivity and also improves peak symmetry by reducing adsorption effects. However a disadvantage is that it is more complicated to use than non-temperature programming.

When preparing a temperature program, the heating and cooling capabilities of the chromatograph heating system must not be exceeded. For example, in the Beckman GC-4 (5), up to 200°C. a temperature rise of 50°C per minute is well within limitations; from 200°C to 300°C, a 30°C per minute rise is satisfactory etc. The GC-4 may be cooled from 500°C to less than 100°C in approximately three minutes if the air dump module is used.

2.8 Flow Control

To regulate the flow of carrier gas, the Beckman GC-4 (5) uses the Dual Flow Controller. This module maintains the carrier gas at an

operator set flow rate over a range of 1 to 500 cc per minute, with a typical value being 60 cc per minute. Regulation of carrier gas flow is necessary for temperature programmed systems because column pressure drop changes with temperature, and this change influences flow rates that in turn effect detector response. The flow rate of carrier gas is maintained within 1% when the ratio of module inlet to outlet pressure is greater than two.

2.9 Programmed Flow Control

The Perkin-Elmer flow programmer (6) accessory is a pneumatically controlled system which permits the pressure along the column to rise exponentially, between preset limits, during a predetermined time interval. The controlling component is a pneumatic, differential flow valve. This valve is arranged to permit column inlet pressure to be returned to regulator pressure by means of a time delay element. The time delay is achieved by the use of interchangeable capillary tubes of various lengths, permitting a number of different program times to be obtained readily, ie. a first order dynamic resistance-capacitance pneumatic system, which produces an exponential response to a step input disturbance. The Flow Programmer is connected between the carrier-gas supply and the inlet of the chromatograph. Any flow or pressure controllers should be by-passed, because inlet column pressure must now be controlled by the Flow Programmer itself.

The advantages of flow programming are:

1) Analysis time is shortened for a wide range of mixtures.

2) Lower operating temperatures can be used, allowing wider choice of the column liquid phase and separation of some thermally-unstable samples.

3) Little base-line drift is observed compared to temperature

programming. This is because, at a given temperature, the bleeding of a column increases only linearly with the column flow rate while, in temperature programming, column bleeding increases exponentially with column temperature.

4) Broad peaks, which would normally emerge later, can be made sharper. This would allow better detection of small concentrations.

5) Flow programming can increase the sample capacity. This was demonstrated by Scott (6), who describes the use of a flow-programmed short analytical column, which permits a sample charge of 175 microliters and still provides useful component separation.

Having now discussed the hardware features of a chromatograph and their various methods of use, the following chapter will attempt to use these specifications in the design of a general chromatograph-computer system.

3. SYSTEM DESIGN OF A GAS CHROMATOGRAPH MONITORING PROGRAM.

The basic problem to be solved by a GC monitoring program, using a time shared computer, can be described in terms of its input and output, as shown in Figure 3.1 (9). The primary input is simply the voltages from the different gas chromatograph detectors and the primary output is the typed analysis reports. Other input requirements are chromatograph start-stop signals, analyst entries, and data required to define the different analysis methods to the system. Other output requirements are contact closures to operate lights and chromatograph valve switching.

The major software objectives in programming a gas chromatograph system are outlined below:-

1) The system should be capable of monitoring several chromatograph detector outputs and make the necessary range changes, if this is under computer control.

2) The system should be capable of identifying and distinguishing the start ups of the different chromatographs.

3) The system should allow the analyst to make analysis entries through some convenient input device such as a typewriter keyboard.

4) The system should be capable of controlling the gas chromatograph hardware such as switching valves etc.

5) The system should be capable of handling both the 'real-time data processing' technique and the 'storage of all the raw data for later processing' technique.

6) A control routine should be provided, which would enable the user to change control parameters, such as smoothing coefficients,

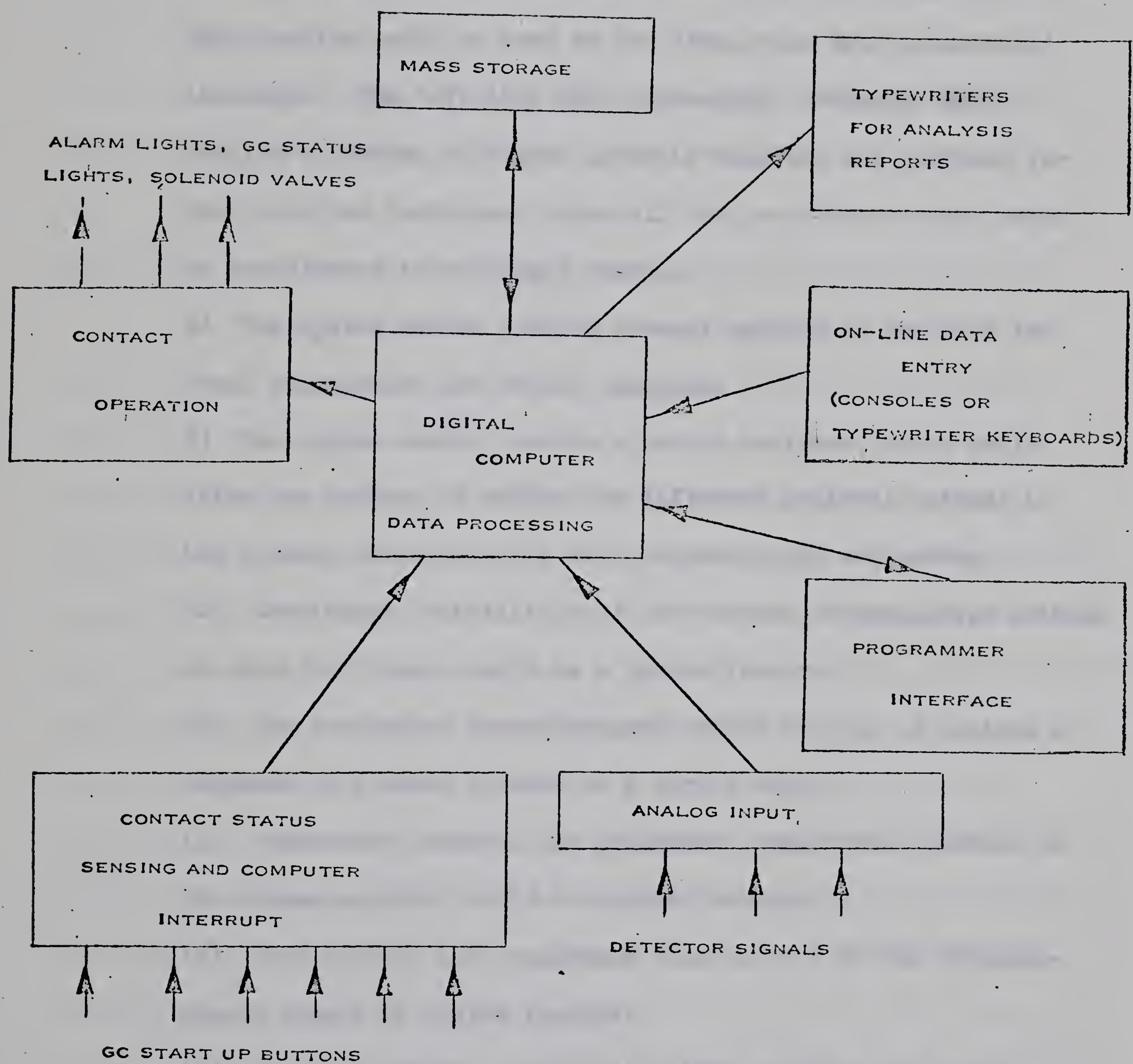
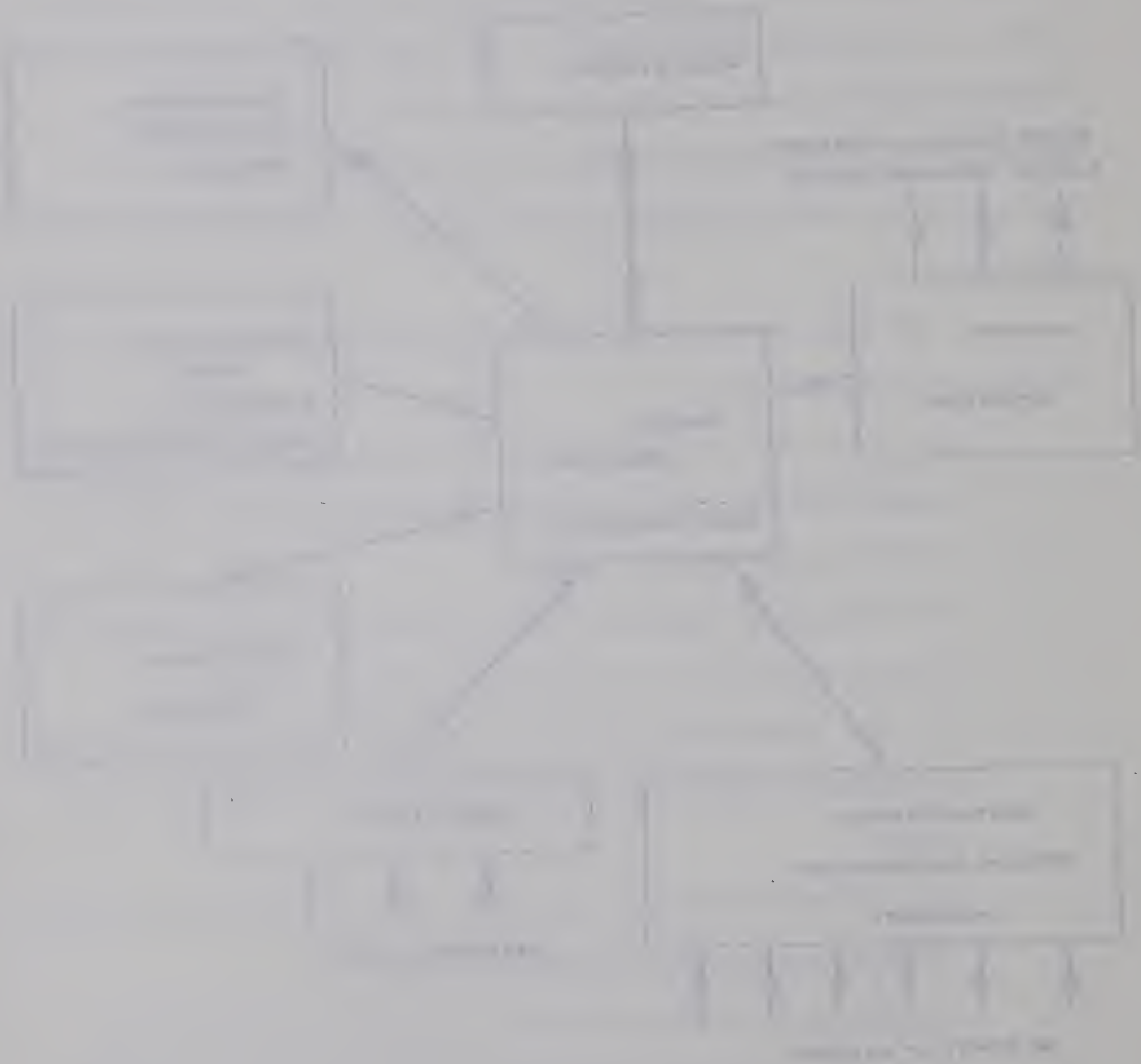


Figure 3.1 Software Objectives Diagram



in the peak detection routines at any time throughout an analysis run.

7) The system should have a routine which transfers all the peak results, detected during the analysis run, from a core output buffer (core storage area) to auxiliary storage files. This routine would be used by the 'real-time data processing' technique. The 'off-line data processing' technique would require a routine of higher priority than the one provided for the real-time technique, since all the raw detector data would be transferred to auxiliary storage.

8) The system should provide several options to the user for final calculation and output routines.

9) The system should provide a set of routines, which would allow the analyst to define the different analysis methods to the system, simultaneously with chromatograph monitoring.

10) Continuous reinitiation of the various chromatograph methods at specified times should be a system feature.

11) The continuous chromatographs should be able to analyze a sequence of process streams on a cyclic basis.

12) Temperature control and programmed temperature control of the chromatographs should be system features.

13) Flow control and programmed flow control of the chromatographs should be system features.

14) An alarm system, capable of either printing out alarm messages or turning off the analyzer power, should be an added system feature. This would be desirable to protect the chromatographs against abnormal operating conditions, such as excessive

current to the detector filaments and other conditions, discussed in Chapter 2.

These various objectives will now be discussed more specifically under the following headings:

1) Data Input/Output between the computer and the chromatographs.

2) Data processing techniques with their computer priorities.

3) Input/Output communication between the computer and the analyst.

3.1 Data Input/Output Between the Computer and the Chromatographs (9)

During the course of a single chromatogram, the chromatograph output voltage from typical detectors may range from only a few microvolts to as high as two or three volts on the larger peaks, a dynamic range of over one million. The computer must use variable range input amplifiers or attenuators to be capable of reading this data with adequate resolution and accuracy, throughout this wide dynamic range. The sampling rate of the detector voltages must be high enough to detect peak occurrences, and then accurately calculate the areas under these peaks. If the maximum number of chromatographs which must operate simultaneously is large, the total sampling rate is usually too high for low speed relay multiplexers. Finally, to make the problem even worse, there is the requirement that little noise be added to the signal as it is multiplexed and converted to a binary or digital number.

Having presented this problem, specific topics listed below will be discussed.

1) Sampling rate considerations

2) Varying vs constant scan rates

3) Filtering

4) Automatic amplification range selection

5) Computer control of chromatographs

3.1.1 Sampling Rate Consideration.

The rate at which the analog input equipment must operate is a function of the speed required for each chromatogram, and the number of chromatographs that are to be attached to the computer.

IBM state (10) that statistical studies of the number of data points, required to calculate a peak area to 0.1% accuracy, have shown that a minimum of eight points per peak is required. Dividing this by the number of seconds that a peak persists will result in a sampling rate. Fast peaks rise and return to the baseline in a second or less. These two requirements dictate that a chromatograph should be sampled at an approximate rate of 10 samples per second. With conventional relay type multiplexing units, the maximum total sampling rate is approximately 100 points per second. Therefore, the number of chromatographs that the system could communicate with simultaneously is dependant upon the sampling rate. For example, with the relay multiplexer unit and a sampling rate of 10 points per second, only 10 chromatographs can operate simultaneously. High speed multiplexing units, capable of handling several thousand points per second, are available for systems which require higher input rates.

3.1.2 Varying vs Constant Sampling Rate.

Obviously, a variable sampling rate for each chromatograph would be a better feature than a constant sampling rate for all the chromatographs. This would allow the sampling rate to be changed at any time during a chromatograph analysis, thereby enabling the user to utilize computer time more efficiently. For example, for a fast eluting peak

the user could specify the maximum sampling rate available, while on a slow eluting peak, a lower sampling rate could be used.

However, this additional feature creates extra programming problems as outlined below:

1) the data input timing program would be more complicated than the simple sampling of a chromatograph point after a fixed time interval.

2) the feature of the fixed time interval, providing a constant interval along the time axis for real time integration of peaks, would be lost.

3) a more sophisticated integration routine would be required along with more complicated baseline correction programs.

3.1.3 Filtering.

The signals which the computer receives from the chromatograph are subject to noise. To be more specific, they can contain voltage spikes that appear to the program as peaks in their unfiltered state. The filtering of these signals is accomplished by a combination of electronic circuits and digital programs.

Some desirable features of these digital filters are outlined below:

1) Exponential smoothing of the first and second derivatives with adjustable smoothing coefficients, to allow changes to be made at any time during the chromatograph analysis.

2) Dead bands around the first and second derivatives, to insure that true signal trends persist and are not the result of noise during a chromatograph analysis.

3) Capability to vary a parameter, which specifies the number

of readings that are to be taken, before the computer makes a firm decision that a slope of the curve is really changing during a chromatograph analysis.

4) Calculation of the first and second derivatives using a least squares technique to minimize high frequency noise.

3.1.4 Automatic Amplification Range Selection (9).

Several methods of obtaining automatic amplification range selection are presented below. Method 1 is shown in Figure 3.2. When this scheme is used, multiple amplifiers are wired in parallel to each chromatograph's output. The high level amplified signals are multiplexed into the analog-digital converter (ADC) by a high speed solid state multiplexer. Switching the signal after amplification minimizes the signal to-noise ratio. Being able to multiplex high level signals means that the multiplexer can be high speed and single ended. This scheme solves the three major problems:

1) reading detector outputs over wide dynamic ranges.

2) multiplexing the analog signals into the ADC at a very fast rate.

3) minimizing noise addition to the signal required.

The wide dynamic range of the chromatograph's signal is handled by switching to the appropriate amplifier under computer control, thereby keeping the input signal at the desired level. However, the major disadvantage of this scheme is economic. The cost of adding multiple amplifiers, typically four, to each chromatograph is high. However, this is partially offset by the increased cost of the hardware requirements for the other methods. Therefore, from an economic viewpoint, this scheme should be overlooked if total system cost has to be kept as low as possible.

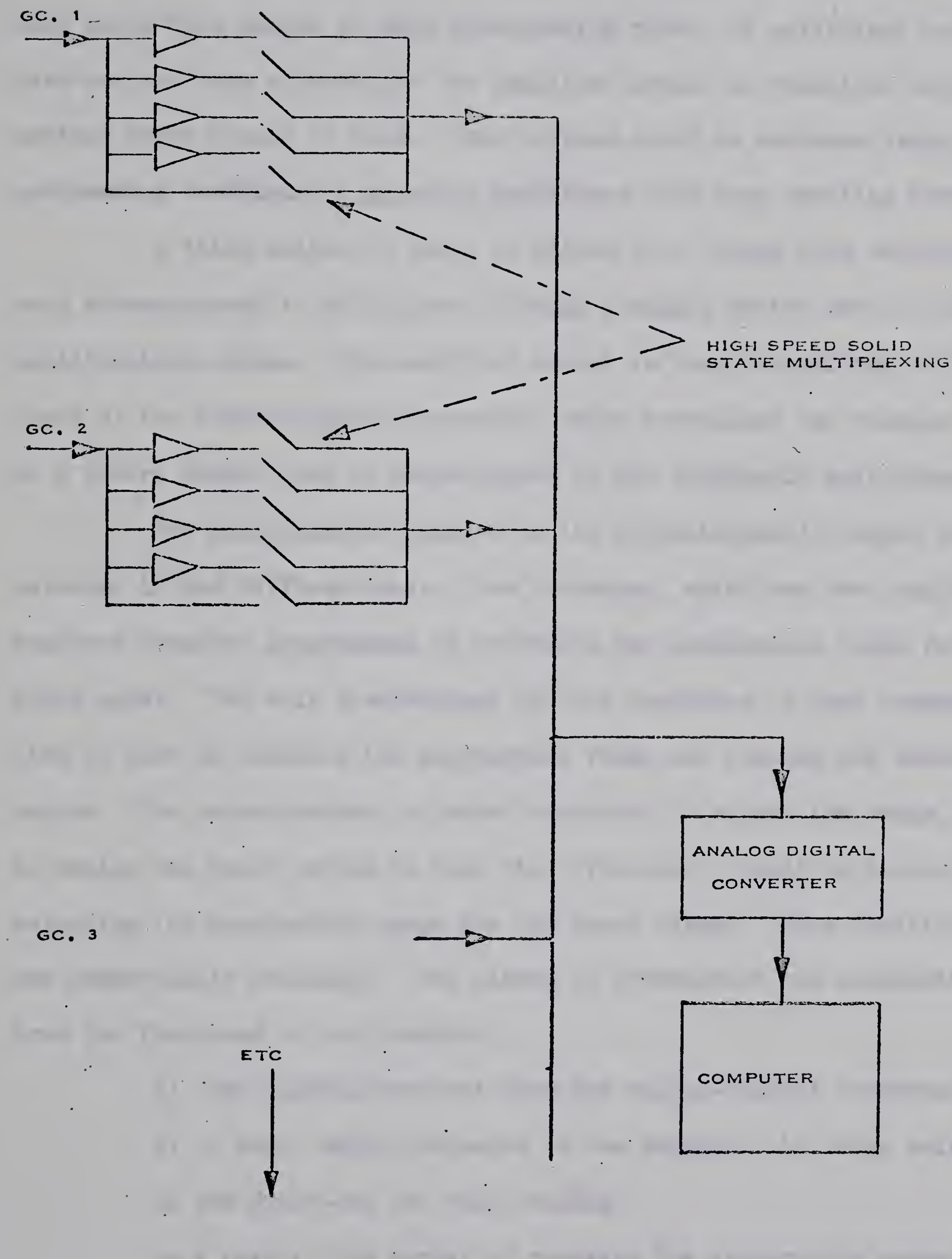


Figure 3.2 Automatic Range Changing Method 1

The second method shown in Figure 3.3 avoids the use of the multiple amplifiers by utilizing a programmed gain amplifier, under computer control, for each chromatograph. Problems might be encountered when using this method at high multiplexing rates, if sufficient response time has not been allowed for the amplifier output to stabilize before another input signal is read. This problem could be overcome using a programming technique or by using amplifiers with fast settling times.

A third method is shown in Figure 3.4. Using this technique, each chromatograph is multiplexed through a single switch into a time shared amplification system. The amplified signal is then transmitted to the input of the analog-digital converter, which translates the voltage signal to a binary number that is proportional to the originally multiplexed signal.

The amplification applied to the chromatograph's signal can be selected in two different ways. One technique, which has been applied, requires computer programming to determine the appropriate range for each input point. The only disadvantage of this technique is that computer time is used in choosing the appropriate range and issuing the switching action. The second method, a better technique to select the range, is to design the input system so that the 'front-end' itself is capable of selecting the appropriate range for the input signal. This facility is now commercially available. Two pieces of information are transmitted from the front-end to the computer;

1) the digital-read-out from the analog-digital converter.

2) a code which indicates to the computer the range selected by the front-end for that reading.

As a result, the burden of choosing the appropriate range is removed from the computer, freeing the computer for other operations. In

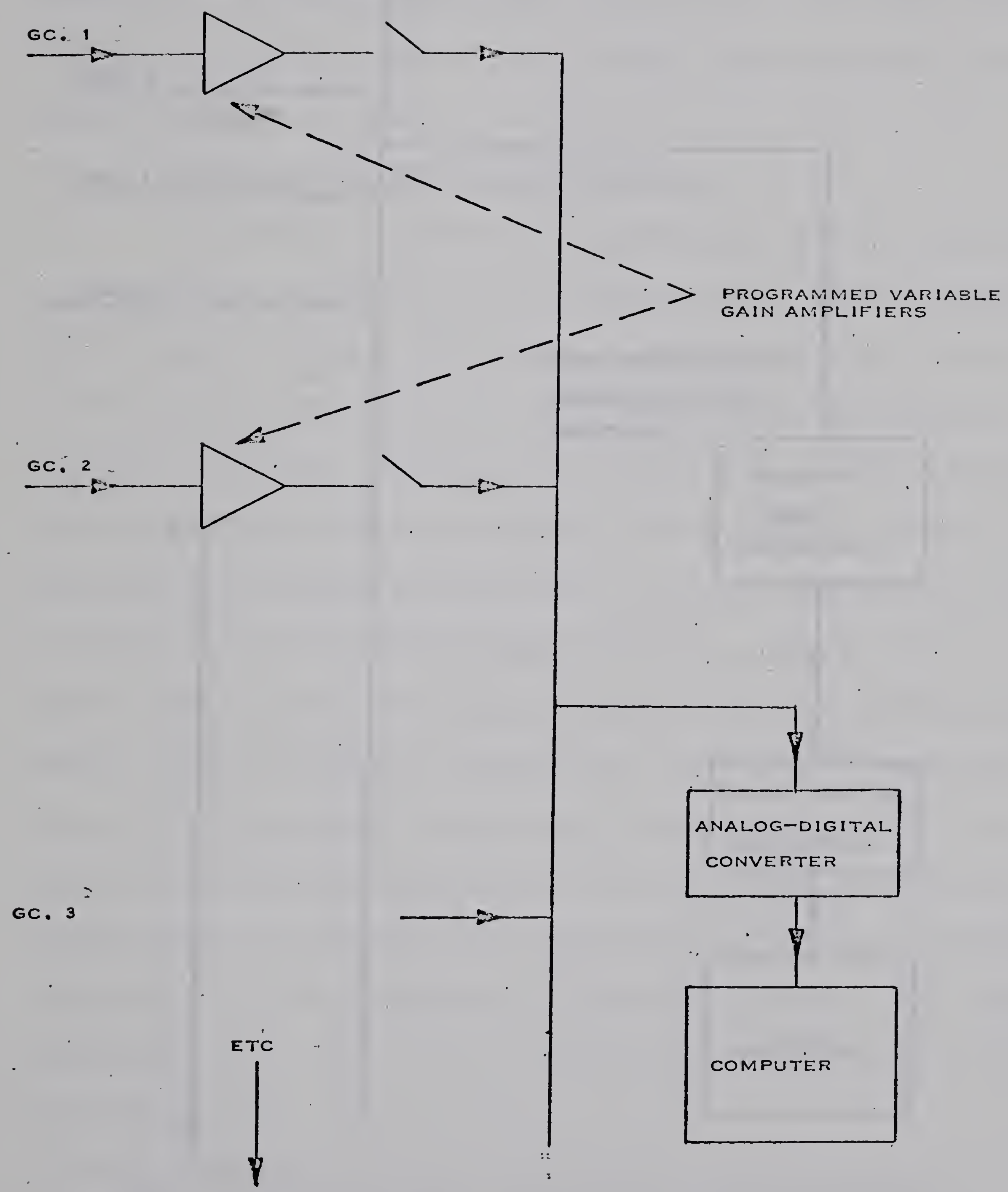


Figure 3.3 Automatic Range Changing Method 2

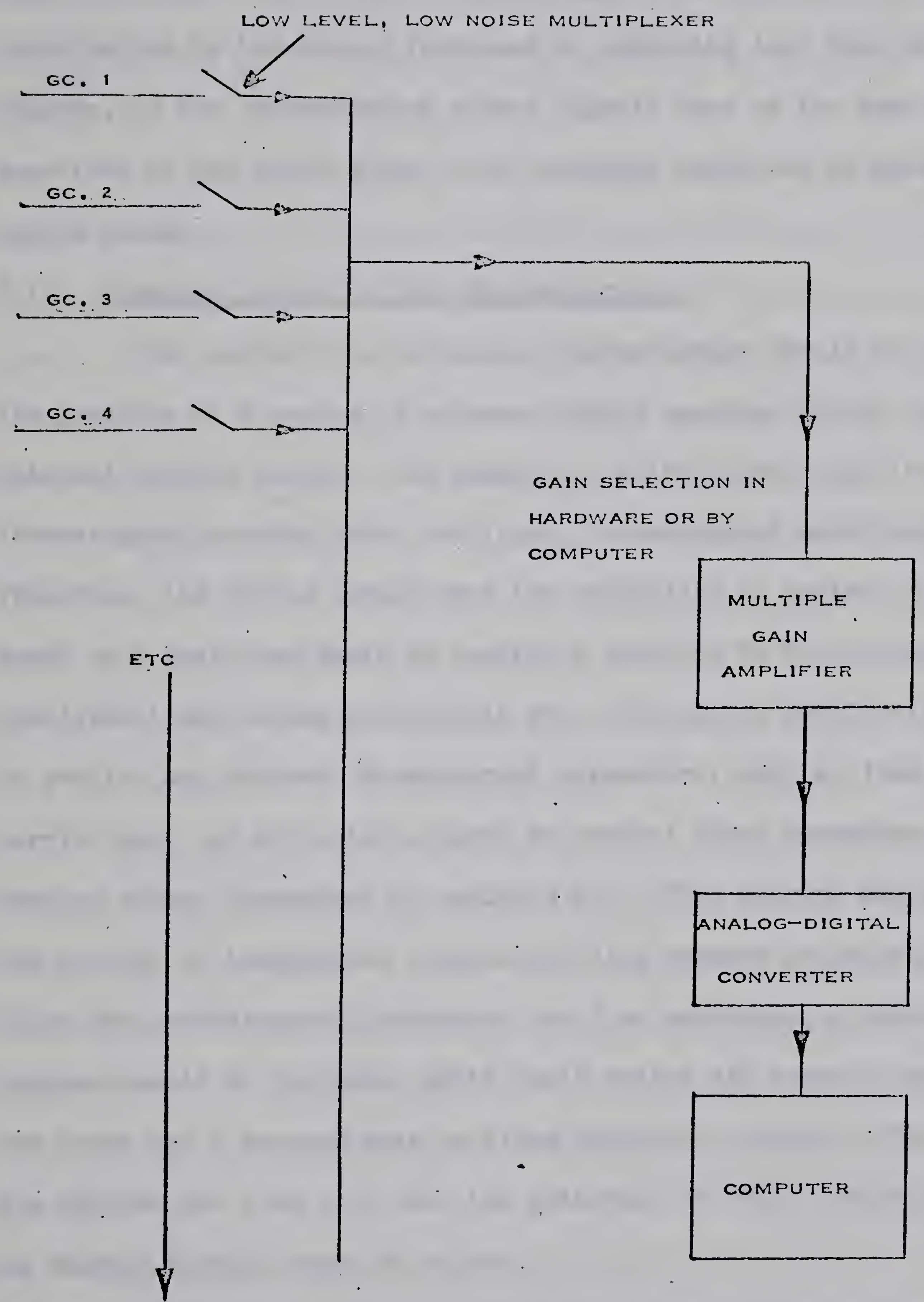


Figure 3.4 Automatic Range Changing Method 3

this scheme, since the signal is multiplexed before amplification, it is absolutely essential that the multiplexer switches have very low noise characteristics. IBM (9) have found that on most amplified ranges, noise contribution by the entire front-end is something less than 10 microvolts. However, if the chromatograph output signals were of the same order of magnitude as the noise value, this technique could not be used in the system design.

3.1.5 Computer Control of the Chromatographs.

The status of a particular chromatograph should be shown near its location by a series of coloured lights operated by the computer external contact points. For example, a white light could indicate chromatograph running and a red light, chromatograph backflushing. Therefore, the system should have the capability to control a chromatograph on a real-time basis by operating switches in the analyzer, at specified times during an analysis run. The system should also be able to monitor any desired chromatograph parameters, such as flow rate of the carrier gas, and allow the analyst to control these parameters at any desired values throughout an analysis run. This program would then allow the analyst to temperature program and flow program an analysis run. Since the chromatograph parameters could be monitored, a protection program should be included, which could switch off power to an analyzer and print out a message when an alarm condition occurred. For example, low carrier gas flow rate over the detectors or other conditions discussed in Chapter 2 would cause an alarm.

3.2 Data Processing Techniques with Their Computer Priorities.

It is assumed that the time shared computer for this application has a series of interrupt levels, which the GC routines can use.

The analyst can then specify computer priorities for his programs. This feature enables higher priority programs to interrupt lower ones presently being executed. Upon complete execution of the higher priority programs, the system will then continue with the execution of the lower priority programs.

To take advantage of this facility, the GC routines can be considered to operate at four interrupt levels, as shown in Figure 3.5. The input routines, because of high frequency of operation, must be given a high priority level. Any programmed range switching must also be performed at this level together with any control actions specified for an analysis run, such as chromatograph valve switching.

Having collected the raw data in a core buffer, (temporary core storage area) either a peak detection program operating at a lower interrupt level can be called to process the data, or a program called which stores all this raw data in auxiliary storage for later processing. The advantages and disadvantages of these two techniques will be discussed in section 3.2.1. Using the real time data processing technique, the chromatogram results are then transferred to auxiliary storage from a core output buffer, as each peak is detected. At the completion of the analysis run, the final calculation and report output routines are executed. No attempt will be made in the following sections to give a lengthy detailed discussion of the individual programs which accomplish chromatograph monitoring and control.

3.2.1 Real-Time or Off-Line Data Processing.

The decision whether to use the real-time or off-line data processing technique is dependent upon each user's application. If the application warrants a completely dedicated computer to monitor chromato-

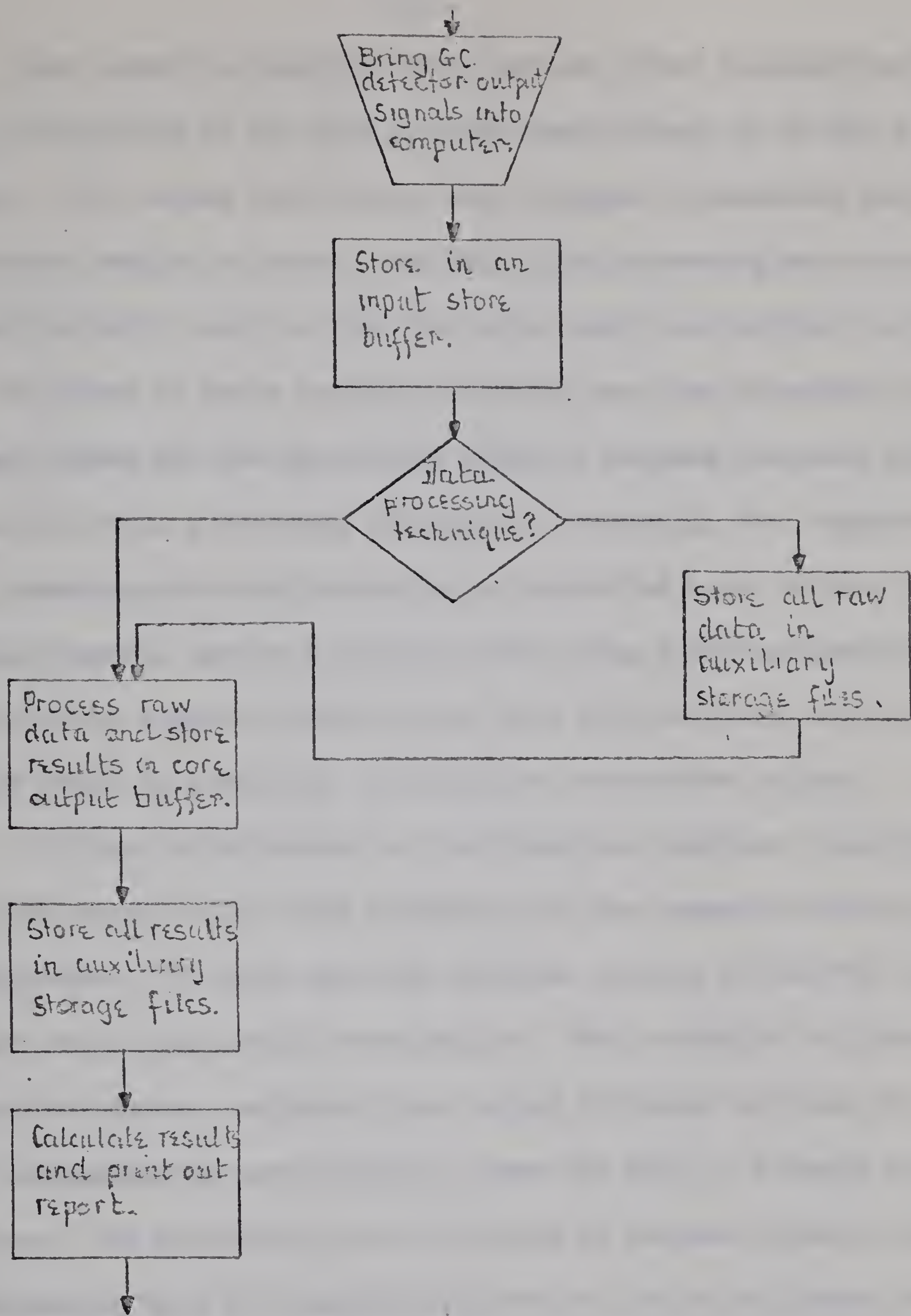


Figure 3.5 Computer Priorities of Data Processing

graphs, then since the computer would have no other calculations to perform, processing of the data on-line would appear to be the better technique. The reason for this is that storage is minimized and computer time between samples is used. The real-time processing must occur at a very high priority level so that the data input core buffers do not overflow. The sizes of these buffers, normally two, are dependent upon the data input speed and the processing speed at maximum computer system load. The real-time data processing technique is essential for computer control of the chromatograph valve switching at specified times during a run. If a feedback control system is desired, utilizing a chromatograph as the detector in the process control loop, then the real-time data processing technique would be desirable to eliminate extra time delays.

If the installation is a multipurpose computer, real-time processing of the data might be too time consuming for the computer during periods of maximum load. In this case the complete storage of all the raw data for later processing would be desirable. This technique is also desirable for fast repeat analysis checks using different defined jobs, when complex chromatograms are involved, since the data is already available in storage. The processing can also occur at computer speed, rather than being dependent upon the analog input rate of the multiplexing unit. With this technique, the initial transfer of raw data to bulk storage from the core buffers must occur at high computer priority level. However, the actual data processing routines can be executed at a lower priority level, which would allow more chromatographs or other jobs to be handled. Since processing speed is no longer critical, a more elaborate integration procedure could be used with the off-line technique which might then require less data to attain the same accuracy.

From the above discussion, it is reasonable to suggest that a system, which has both these data processing techniques, would be the best solution for a time shared computer. However, this would cause additional complications in the programming and also utilize extra storage both in core and on disk, thereby increasing the cost factor.

The above system design was considered using various data processing techniques after the raw chromatograph detector output signal has been read into the computer. Another GC system design (11), shown in Figure 3.6, utilizes separate digital integrating units with each chromatograph and a small digital computer. The digital integrating unit detects the peaks, integrates the peaks to determine the areas and corrects for baseline drift independent of the computer. Each time a peak is detected, the peak data is read into the computer and the integrator reset for the next peak. This method eliminates computer data processing of the raw detector data, and only uses the computer to perform the calculations and printout the results. This method certainly provides the user with a good back up feature on computer failure. However, the real-time analyzer valve switching feature and the control parameter changing feature, discussed in the other system design, would be lost. In addition to this, the cost, $7,000 per unit being typical, is rather high.

3.2.2 Control Parameter Changes.

The analyst should be able to define chromatograph jobs so that changes in the control parameters, such as smoothing factors and sampling rates, can be specified to occur at any time during a chromatograph run. Therefore, to have this capability, there would have to be control routines which provide a control check each time a raw data

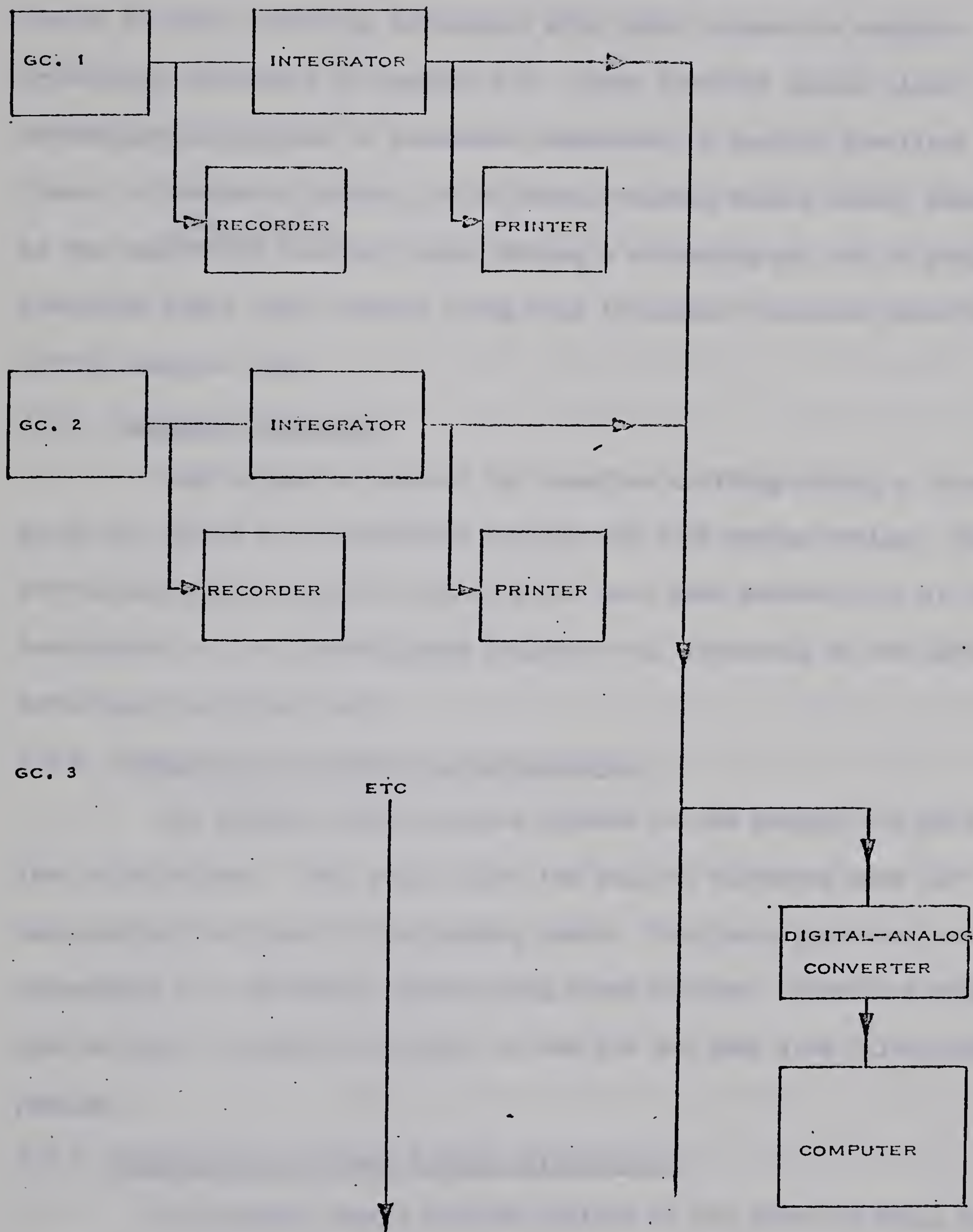
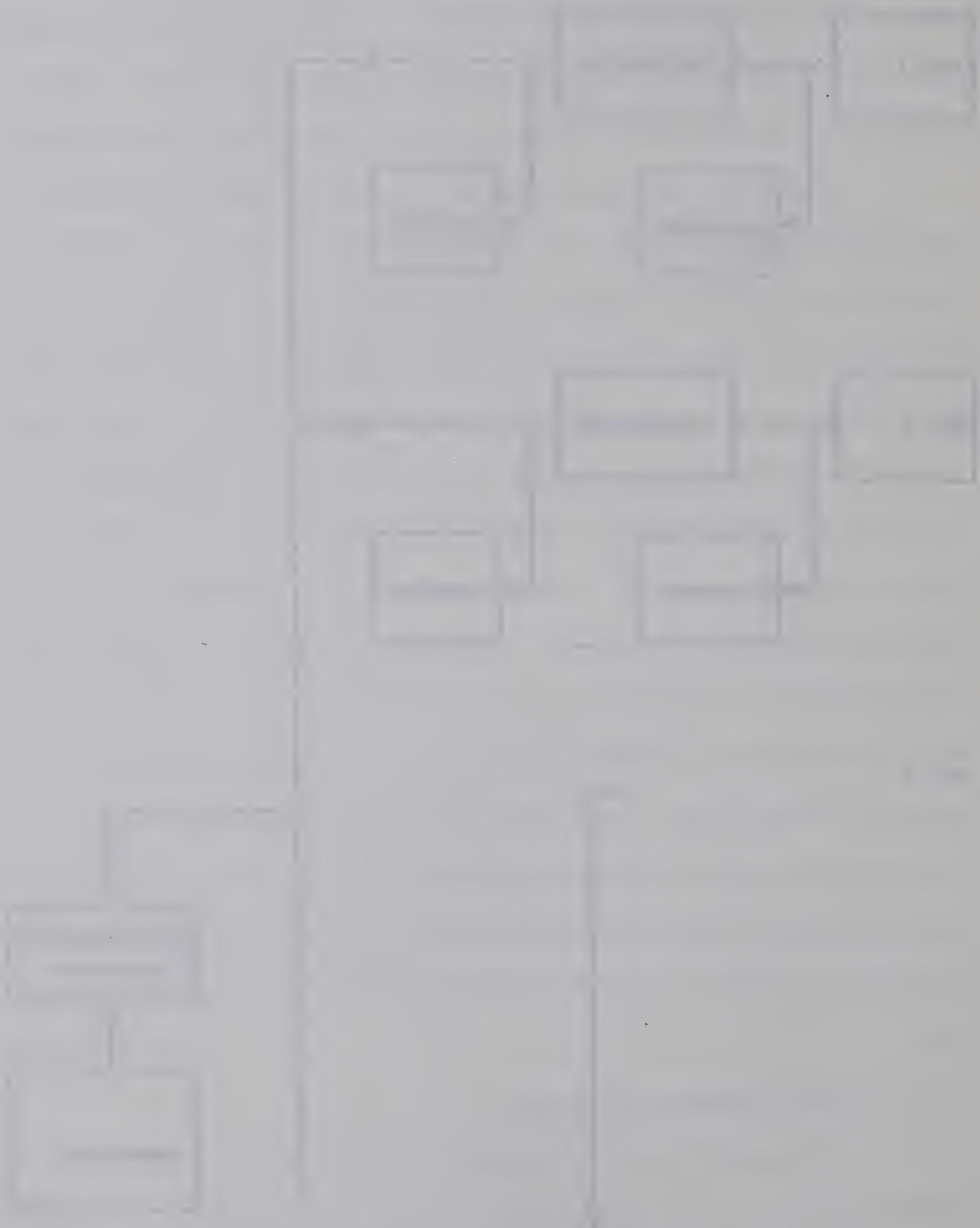


Figure 3.6 Hardware Configuration and Block Diagram for a Multiple Integrator System



point is processed, and make the desired changes at the analyst specified times. These routines are not affected by the user's choice of data processing techniques with their respective computer priorities, discussed in section 3.2. These routines should allow chromatograph analyses to terminate themselves at analyst specified times. A desirable feature, which these routines should allow, would be the capability to detect peaks during a chromatograph run at analyst specified times only, thereby being able to neglect unwanted peaks and saving computer time.

3.2.3 Baseline Correction

Some method to correct for baseline drifting during a chromatograph run should be an essential feature for this system design. This correction could be applied either after each peak detection or at the termination of the chromatograph analysis run, depending on the particular correction technique used.

3.2.4 Flexibility of Peak Area Calculations.

The program should provide options to the analyst for peak area calculations. This would allow the analyst different ways for calculating the areas of overlapping peaks. Thus, each peak area could be calculated in a different manner using these options. Provision should also be made to allow the analyst to add his own peak area calculation routine.

3.2.5 Flexibility of Final Report Calculations.

The program should provide options to the user for final report calculations and printout. This would allow various analyses techniques to be carried out, such as a standardization run and an unknown run. The standardization run would calculate and printout standardization factors,

whereas the unknown run would provide concentration values in the final printout. The program should allow peaks to be combined and reported as one result if desired, such as all the hydrocarbons in a sample reported together. Provision should be made to allow the analyst to add his own final report calculation and formatting routine.

3.3 Input/Output Communication Between the Computer and the Analyst.

The start of a chromatograph analysis could be indicated to the computer by a push button located on the particular chromatograph. The computer should acknowledge this start up signal in some manner, such as switching on a light at the chromatograph, to inform the analyst when to inject the sample into a laboratory gas chromatograph.

To enable the computer to identify components and calculate concentrations, the analyst must specify how each analysis is to be handled and provide the computer with this data. An extensive file system plus maintenance program should be provided to achieve this function, which would allow an analysis method to be referenced with minimum analyst entries, once this method had been defined completely to the computer. The maintenance program should allow technical personnel in the laboratory to define new analysis methods, change old ones or delete an obsolete technique, by merely filling out data sheets. These data sheets should describe specifically how each analysis is to be handled. These sheets could then be keypunched into cards and read into the computer by the file maintenance program. This program should analyze the data, reformat it and store it in auxiliary storage files for later use, when the actual analysis is performed in the laboratory. Documentation of the analysis procedures should now be stored in auxiliary storage so that external documentation and filing of data sheets could be eliminated. A

full print out of the analysis method could be obtained on request from the analyst by feeding a certain control card through the card reader.

The maintenance procedures, just outlined, should have the capability of on-line performance while the computer is monitoring the chromatographs or doing other jobs such as direct digital control. Once these analysis methods are stored in auxiliary storage, the analyst should be able to specify a previously defined method through a suitable input device, such as a keyboard, and the computer obtain the necessary data from its files to perform the specified analysis.

Having now discussed the systems design principles of a general computer chromatograph system, the following chapter will illustrate one of the industrial chromatograph systems, the IBM 1800 Gas Chromatograph Monitoring Program.

4. THE IBM GAS CHROMATOGRAPH MONITORING PROGRAM

The IBM GC Program (7) is capable of monitoring a number of research type gas chromatographs using an IBM 1800 Process Control Computer.

4.1 General Program Description.

The features of this program are as follows:

1) Read the analog voltage outputs from the chromatograph detectors and attenuate these signals if required, by changing the amplification ranges under programmed control.

2) Control the valve switching operation on the chromatographs and change the peak detection parameters at analyst specified times throughout an analysis, such as the job completion time.

3) Detect, via the on-line data processing technique, the chromatograph peaks and calculate the area under each peak.

4) Store the detected peak results, such as peak start, finish, area, etc. in the correct disk files for later report calculation routines.

5) Identify the chromatograph peaks upon job completion. Baseline correct the peak areas, apply correction factors, calculate and printout the results on a typewriter simultaneously with monitoring the remaining on-line chromatographs.

6) Accept the following information from the analyst via the keyboard:

a. Job heading code

b. Job number

c. Chromatograph detector number

d. Month, day, time

e. Analyst number

7) Detect the analyst start up and termination of a chromatograph job, via a push button.

8) Allow the user to add new jobs (analysis methods), modify or delete old ones, and pack disk records simultaneously with chromatograph monitoring.

The programs associated with these features are divided into six sections listed below and discussed in the following pages:

1) Cold Start Routines

2) Scan Routines (data input, peak detection and control routines)

3) Scan Data Storage Routines

4) Chromatograph Termination, Calculation and Output Routines

5) Chromatograph Job Initialization Routines

6) Disk Maintenance Routines

A simplified overall flow diagram of the GC program, indicating the various program priorities, is shown in Figure 4.1. This shows the capability of the IBM Time Shared Executive (TSX) to execute high priority routines first, such as the data input routines. The system will even interrupt lower priority routines to perform this task, after which execution of the lower priority routines is resumed. An overall flow diagram of the chromatograph monitoring program is shown in Figure 4.2.

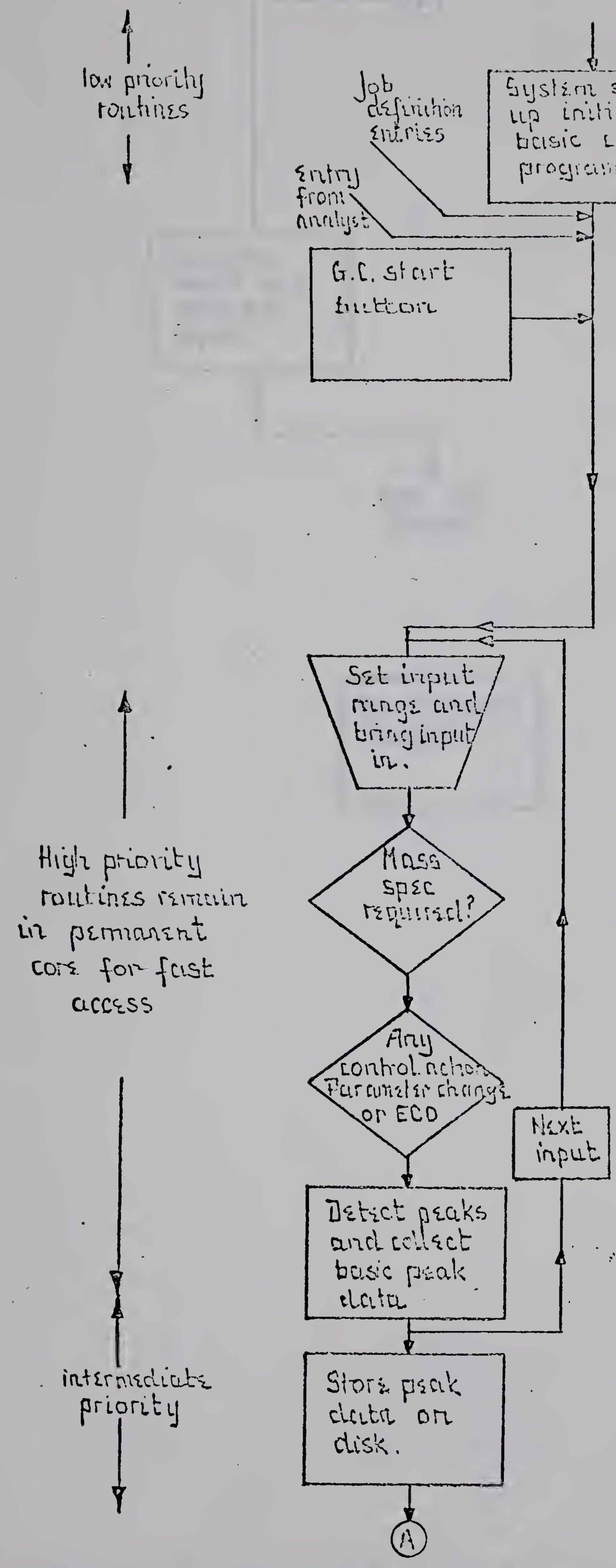
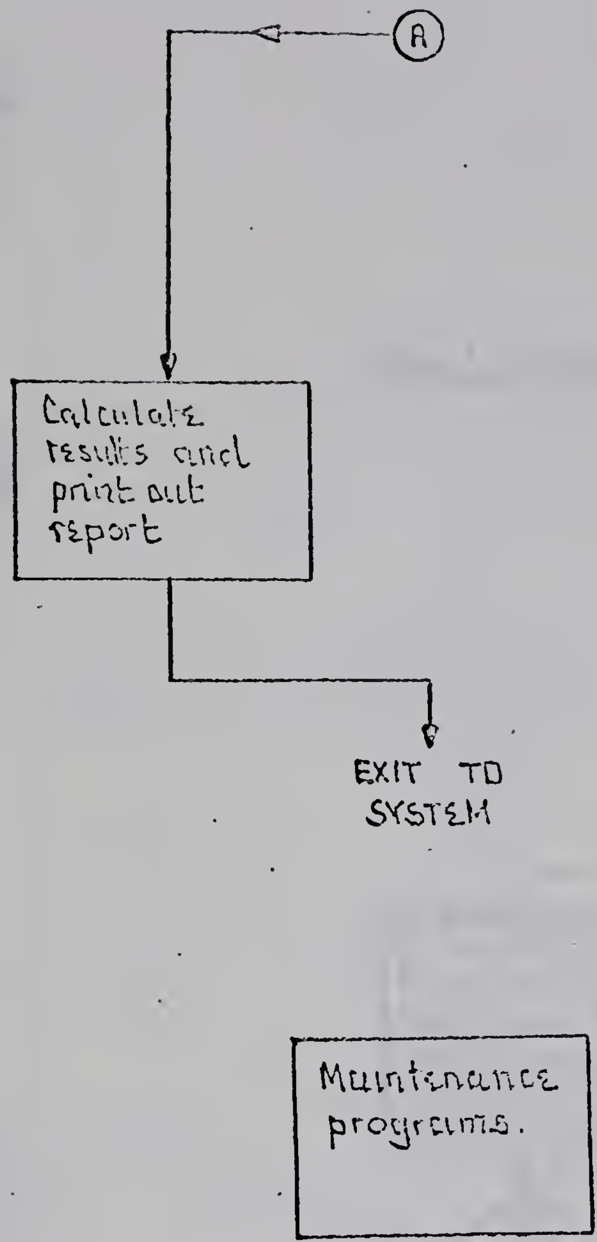


Figure 4.1 Simplified Overall Flow Diagram of the IBM GC Program


A
Calculate results and print out report
EXIT TO SYSTEM
Maintenance programs.

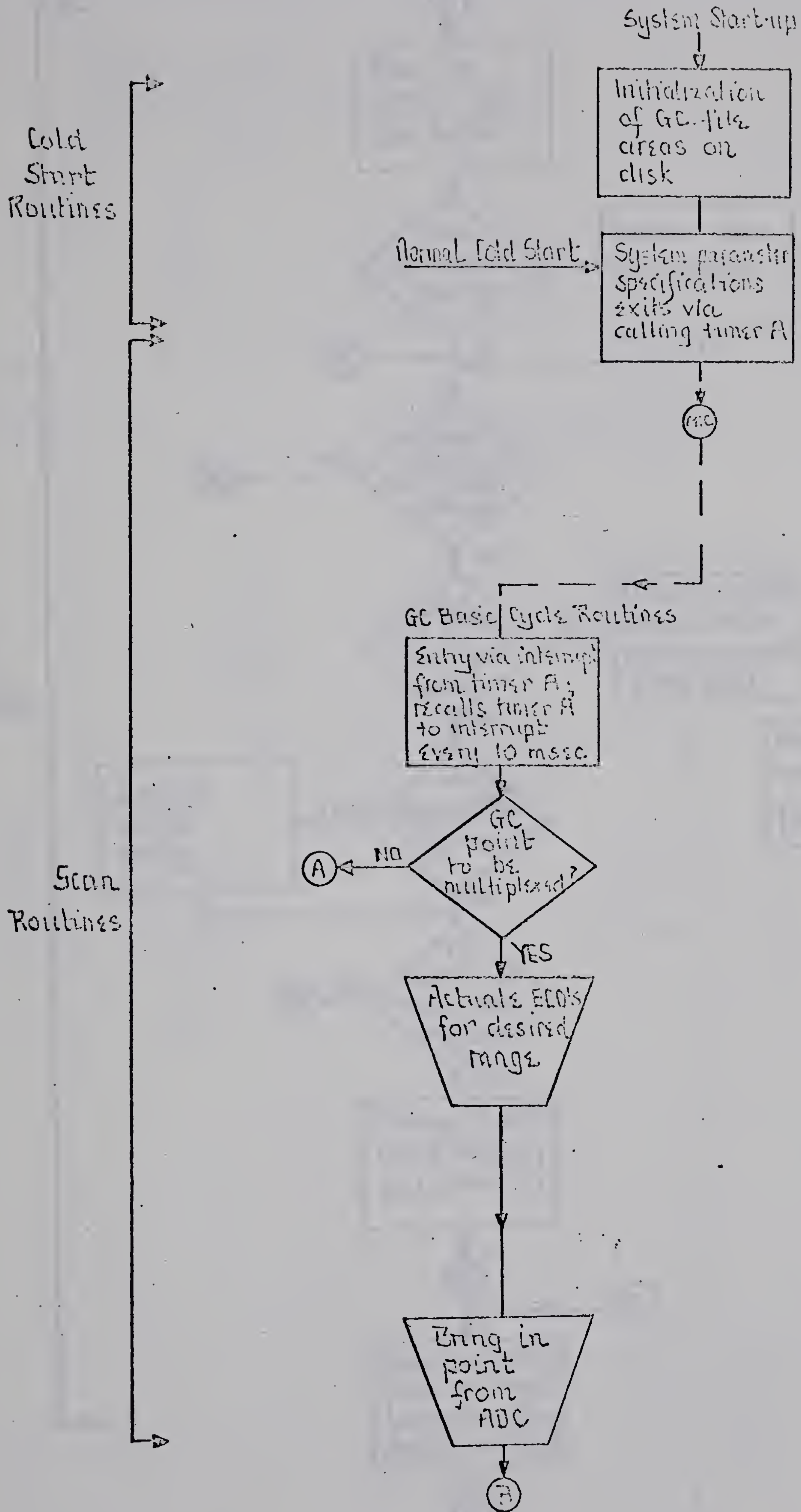
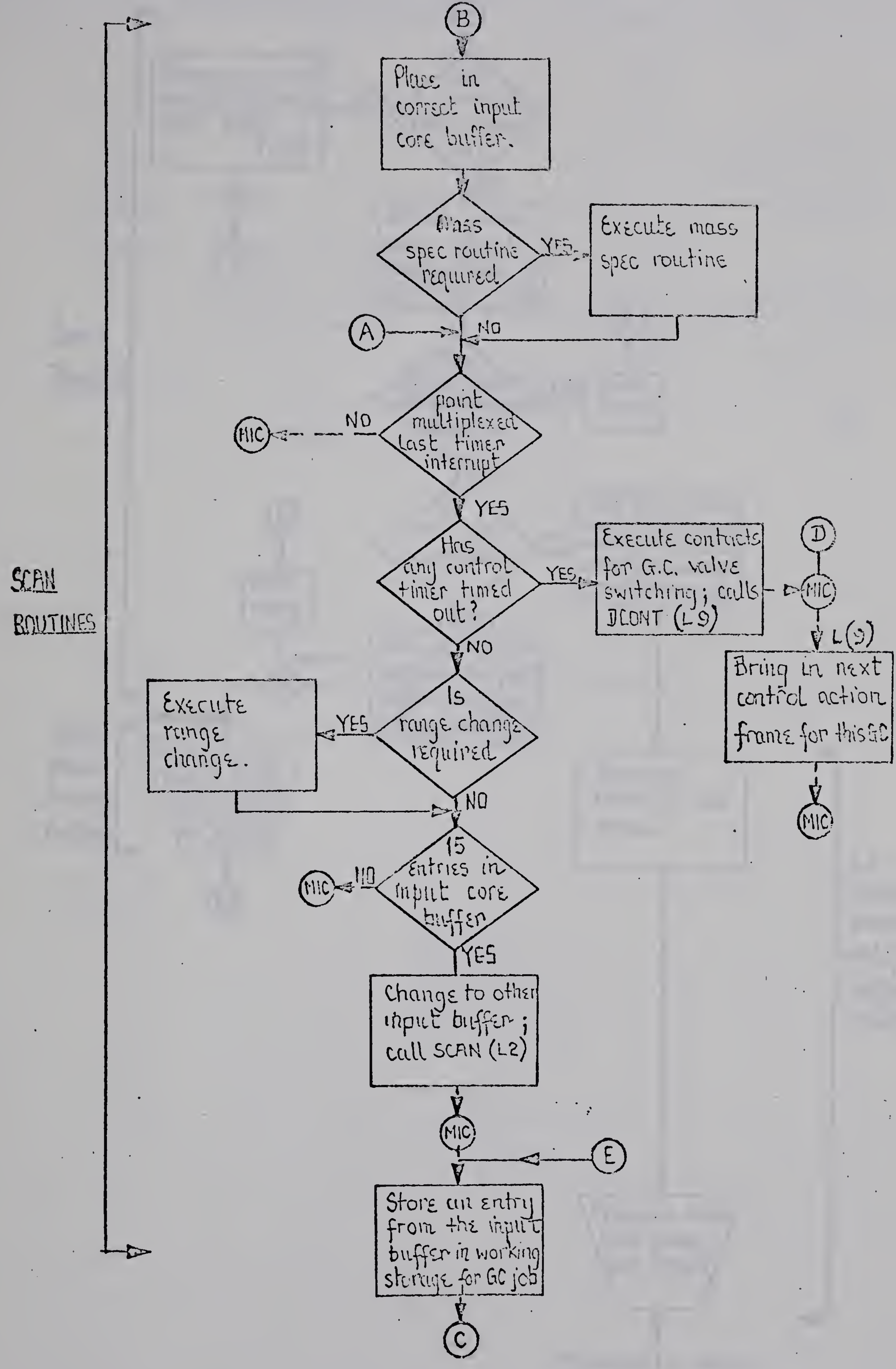
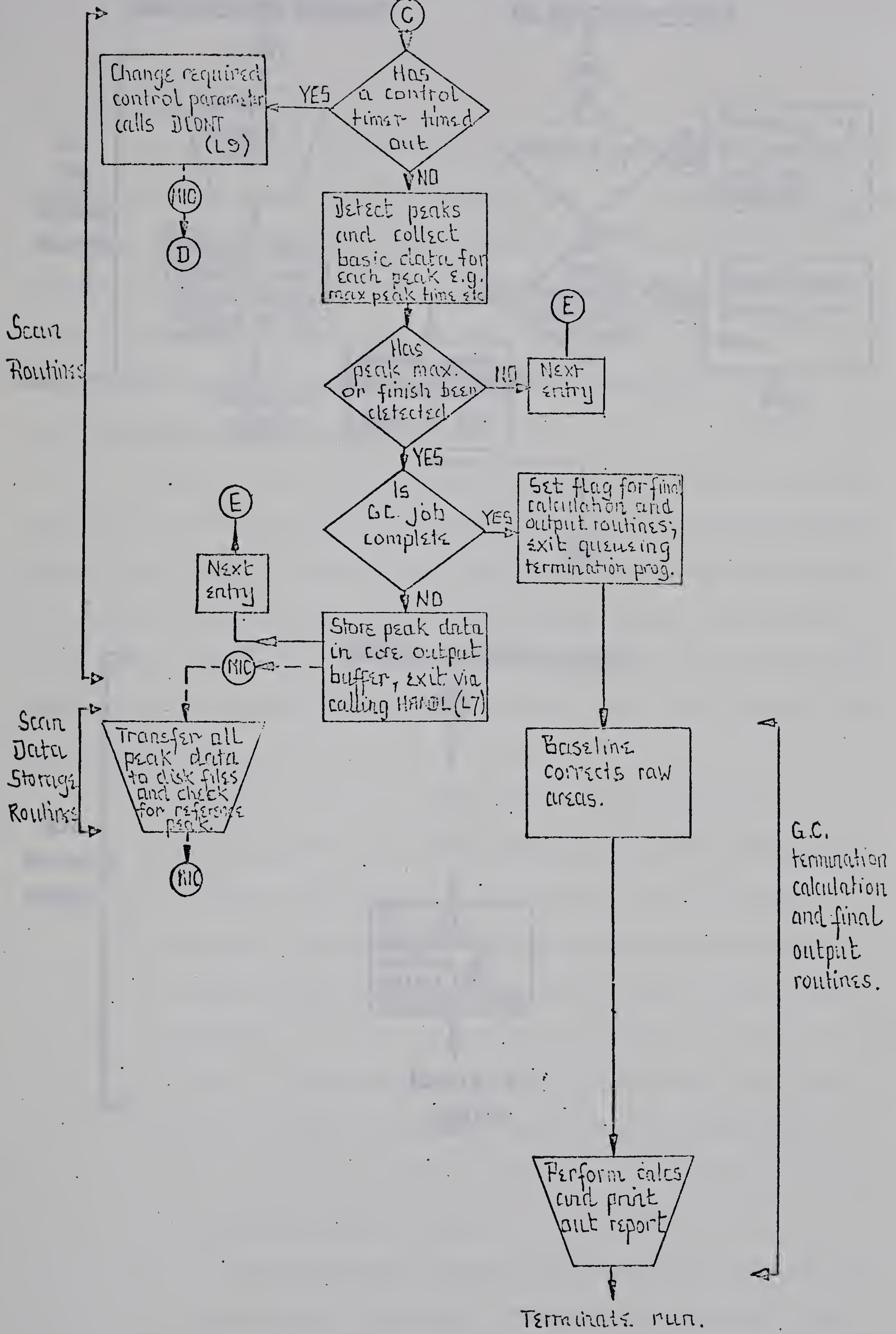
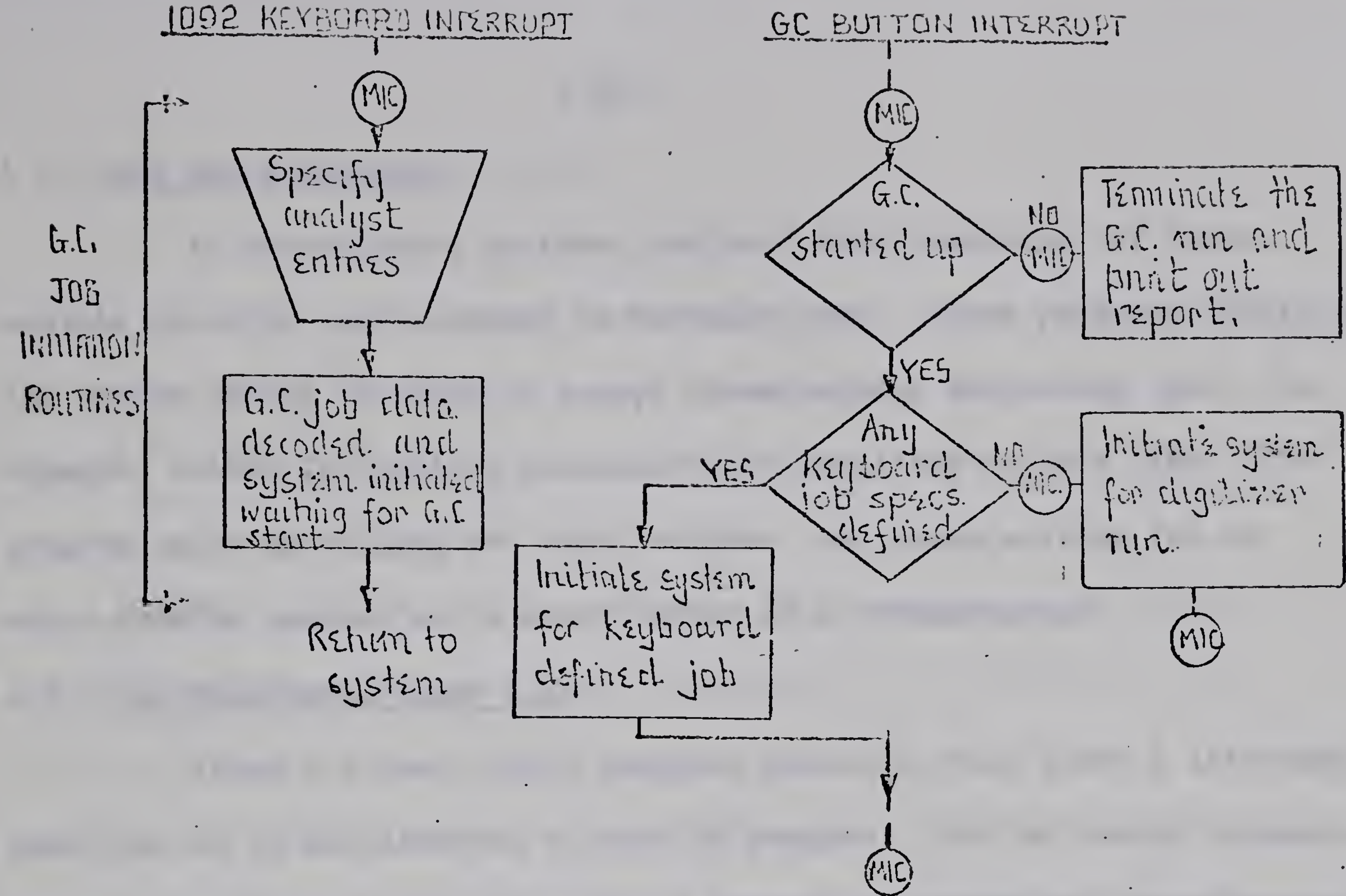
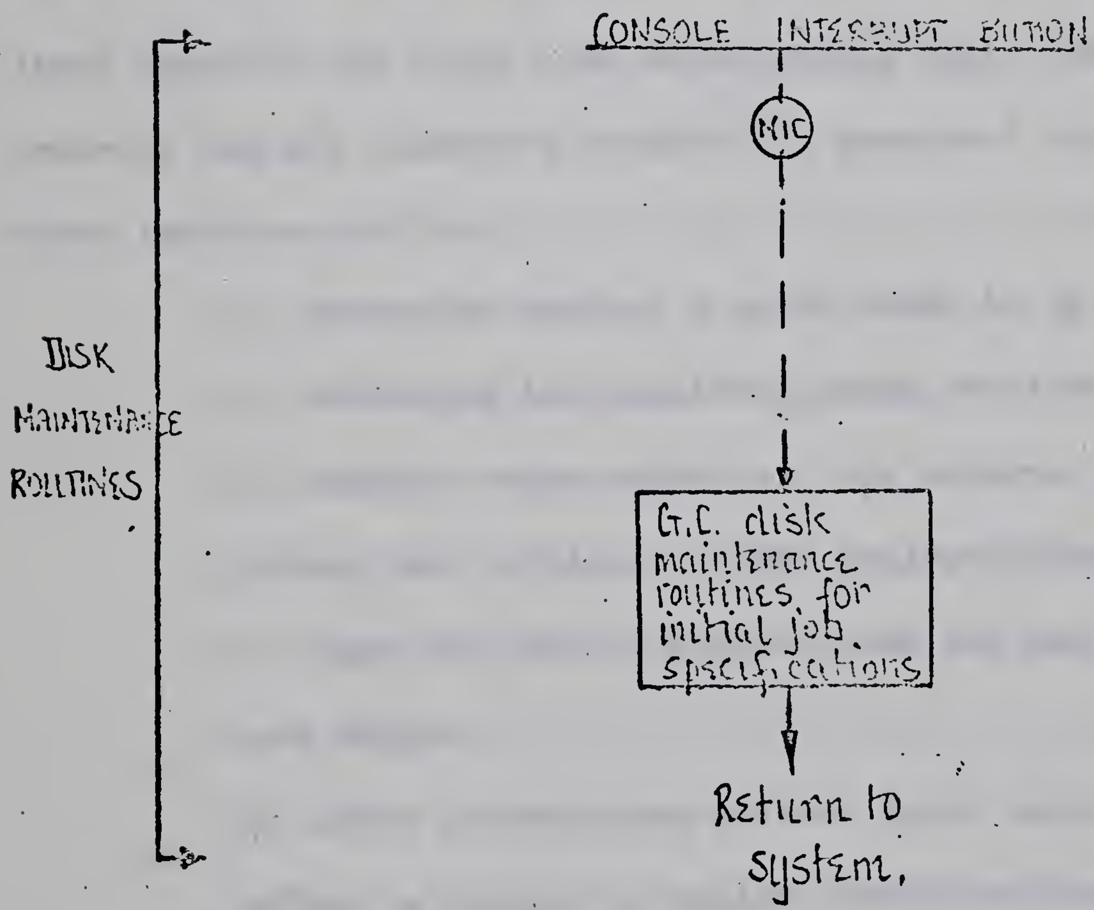


Figure 4.2 . Overall Flow Diagram of the IBM GC Program


B
Place in correct input core buffer.
Mass spec routine required
YES
Execute mass spec routine
A
NO
point multiplexed last timer interrupt
NO
MIC
YES
Has any control timer timed out?
YES
Execute contacts for G.C. valve switching; calls DCONT (L9)
D
MIC
L(9)
Bring in next control action frame for this GC
MIC
NO
Is range change required
YES
Execute range change.
NO
15 entries in input core buffer
NO
MIC
YES
Change to other input buffer; call SCAN (L2)
MIC
E
Store an entry from the input buffer in working storage for GC job
C
SCAN ROUTINES


C
Has a control timer timed out
YES
Change required control parameter calls DCONT (L9)
NIC
D
NO
Detect peaks and collect basic data for each peak e.g. max peak time etc
Has peak max. or finish been detected.
NO
Next entry
E
YES
Is G.C. job complete
YES
Set flag for final calculation and output routines; exit queueing termination prog.
NO
Store peak data in core output buffer, exit via calling HANDL (L7)
Next entry
E
NIC
Scan Routines
Scan Data Storage Routines
Transfer all peak data to disk files and check for reference peak.
NIC
Baseline corrects raw areas.
G.C. termination calculation and final output routines.
Perform calcs and print out report
Terminate run.


1092 KEYBOARD INTERRUPT
GC BUTTON INTERRUPT
MIC
G.C. JOB INITIATION ROUTINES
Specify analyst entries
G.C. job data decoded and system initiated waiting for G.C start
Return to system
G.C. started up
NO
YES
Terminate the G.C. run and print out report.
Any Keyboard job specs. defined
Initiate system for keyboard defined job
Initiate system for digitizer run.


CONSOLE INTERRUPT BUTTON
MIC
DISK MAINTENANCE ROUTINES
G.C. disk maintenance routines for initial job specifications
Return to system.

4.2 Cold Start Routines

At system start up time, the cold start routines, of intermediate priority, are executed in variable core. These programs initialize the system making it ready to accept chromatograph monitoring jobs. For example, values for certain parameters are specified at this time. The program exits by calling the Scan Routines and loops waiting for an entry from an analyst and a start button of a chromatograph.

4.3 Scan Routines (Figure 4.2)

These are basic cycle programs executed every timer A interrupt, specified as 10 milliseconds in this GC program. For the twenty chromatograph system, this is equivalent to 5 points per second per chromatograph or a total of 100 points per second being scanned, which is the maximum input speed of the relay type multiplexing unit. They are extremely high priority and are therefore resident in permanent core. The functions of these routines are to:-

1) determine whether a point needs to be multiplexed.

2) determine the amplifier range required by the analog input.

3) execute range selection, via external contact operate points, and initiate the Nth analog-digital converter input.

4) take the (N-1)th digital read out and place into the input core buffer.

5) after accumulating fifteen input points in the core input buffer, a routine is called, which performs the following:

a. transfers a point into the correct chromatograph working storage area in core,

b. calculates the first and second derivatives, using this point and the last twelve points by a least squares slope

of a line technique,

c. detects peaks using these derivative values,

d. collect basic peak information, shown in Figure 4.3, for each peak,

e. repeat parts a. through d. for each of the fifteen entry points.

The peak detection techniques, using the first and second derivatives, are explained clearly on pages 11 - 15 inclusive of the GC Manual (7).

6) place this basic peak information in the core output buffer and call the storage program to transfer this data from core into the correct disk files.

7) maintain the chromatograph clocks in core (one per chromatograph)

8) check the chromatograph clocks for comparison with the control actions times specified in the job definition, and convert the relative time to absolute time if the reference peak has been detected.

9) execute control actions such as switching contacts at required relative times (relative to the reference peak), to change the flow of gas through the column or to change the peak detection parameters.

10) check for job completion and call final calculation and report routines.

These routines are driven by timer A and the cycle time is chosen by the user in the cold start routines.

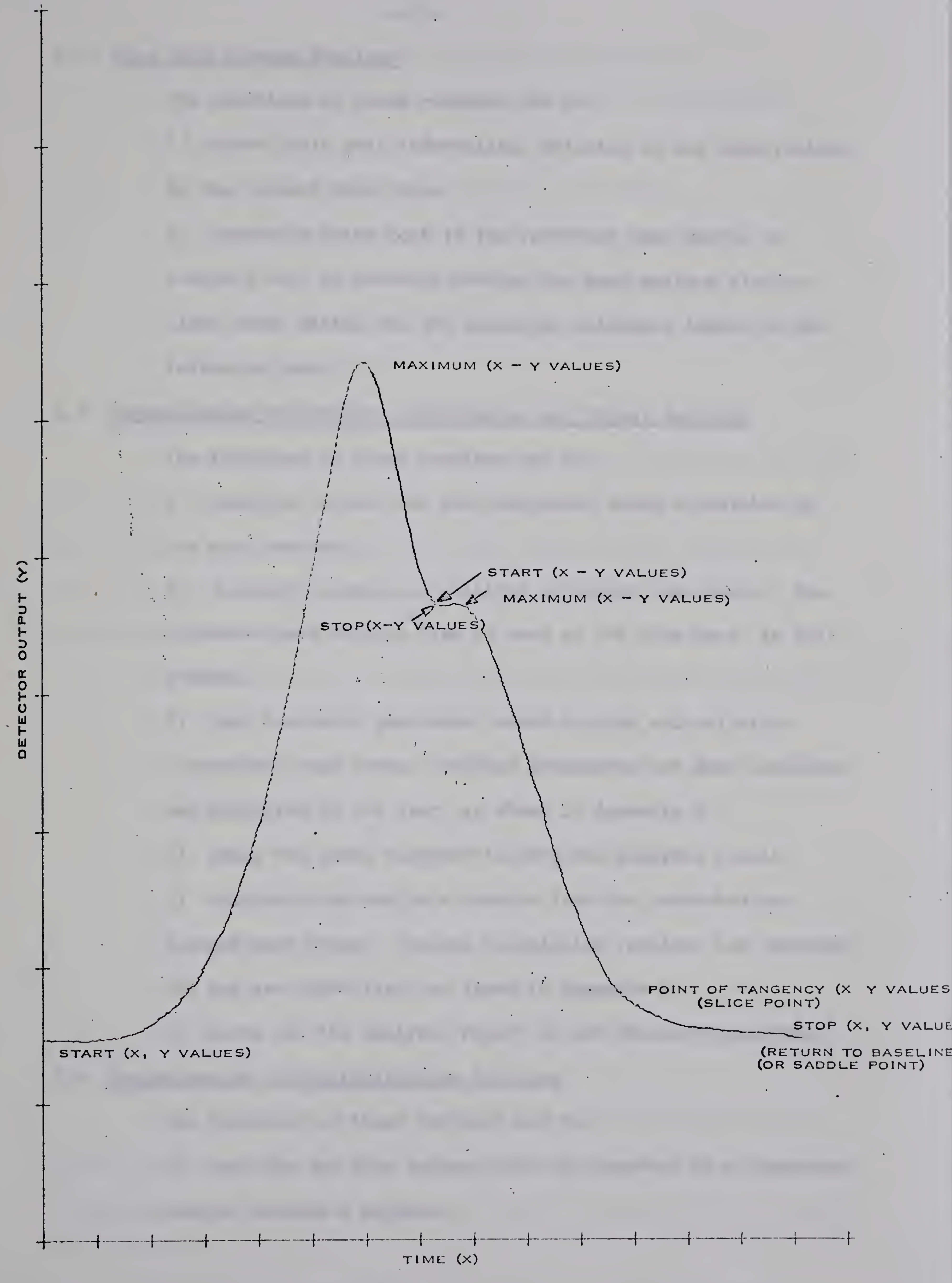


Figure 4.3 Peak Data Detected and Stored by the Scan Routine

4.4 Scan Data Storage Routines

The functions of these routines are to:-

1) store basic peak information, detected by the scan routine, in the correct disk files.

2) determine which peak is the reference peak during an analysis run, by checking whether the peak maximum elution times occur within the job specified tolerance limits of the reference peak.

4.5 Chromatograph Termination, Calculation and Output Routines

The functions of these routines are to:

1) baseline correct the raw integrated areas calculated by the scan routines.

2) identify peaks on a relative retention time basis. The reference peak elution time is used as the time base in this program.

3) take the basic peak data stored on disk and calculate 'corrected' peak areas. Various procedures are made available and specified by the user, as shown in Appendix B.

4) group the peaks together to form one analysis result.

5) calculate the analysis results from the corrected and grouped peak areas. Various calculation routines are provided and are user specified, as shown in Appendix B.

6) print out the analysis report on the desired typewriter.

4.6 Chromatograph Job Initialization Routines

The functions of these routines are to:

1) read the job data entered into the computer by a laboratory analyst through a keyboard.

2) decode this data to determine the following:

a. the job specification to be used in analyzing the sample.

b. the chromatograph detector to be used.

c. the time and date when the sample was taken.

d. the analyst who injected the sample.

3) inform the scan routine to begin accepting data for the sample just injected.

4.7 Disk Maintenance Programs

These routines are executed in variable core and have very low priorities. One function of these programs is to interpret the coded data records, supplied by the user, which define to the computer how each individual analysis is to be monitored. The functions of the maintenance programs are to:

1) define variable values referred to in the job definition.

2) define chromatograph job techniques.

3) retrieve the data from files and present it in an easily understood form.

4) define the meaning of the keyboard buttons.

5) delete old jobs.

6) update old jobs.

7) keep data files packed on disk.

Monitoring of the chromatographs does not have to be terminated in order to execute these programs.

The contents of this chapter outline a chromatograph-computer system in which the computer is completely dedicated to the monitoring of research type gas chromatographs. In order to combine this GC program

with another major system, such as the Direct Digital Control (DDC) program to provide composition control loops for the DDC program, modifications to the original IBM GC program are required. These modifications will be presented in Chapter 5.

5. MODIFICATIONS TO THE IBM GC MONITORING PROGRAM

The original IBM GC Program was written for a completely dedicated IBM 1800 computer application, to monitor twenty laboratory gas chromatographs simultaneously. However, since the Chemical and Petroleum Engineering Department at the University of Alberta has approximately five laboratory chromatographs and one process chromatograph, the GC program was modified and integrated with the Direct Digital Control and Process Operators Console programs, to enable the following functions to be carried out simultaneously.

1) digital control of process loops
2) monitoring of laboratory chromatographs
3) process control using a process chromatograph as the detector in a control loop.

The program modifications will be discussed generally in this chapter, outlining the reasons for modification and the problems encountered attempting to implement them.

5.1 Machine Configuration

Since the University of Alberta has a different machine configuration, shown in Figure 5.1, than the GC program specifications in the GC Manual (7), modifications had to be made. The only major machine configuration difference is that a model 1816 typewriter keyboard replaces two model 1092 programmed keyboards. This allowed the omission of several programs, which service the model 1092 programmed keyboards and interpret the entries made by the analyst. However, new programs were written to service the model 1816 typewriter keyboard and interpret the analyst entries, so that the same connecting programs could be used. These programs are documented in Appendix A.1.

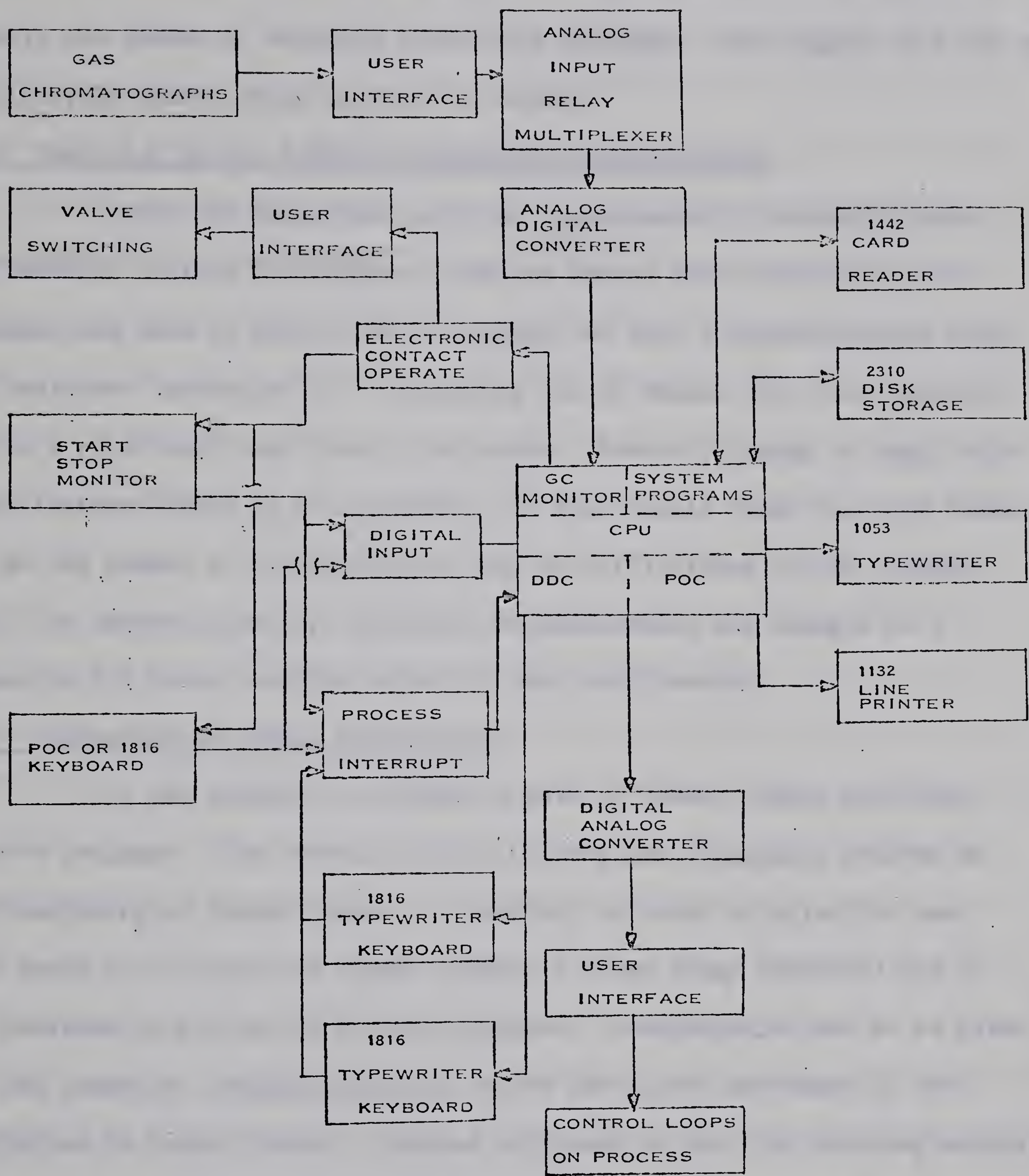


Figure 5.1 University of Alberta IBM 1800 Machine Configuration

At TSX system generation time, 5 cylinders of message buffering between disk storage and the output printers were considered sufficient to handle the number of messages stored for printing. This figure of 5 was an arbitrarily chosen value made by the author.

5.2 Reduction in the Number of Monitored Chromatographs

Since the department only has approximately 5 chromatographs, in order to utilize the computer time and memory more efficiently, an attempt was made to modify the GC program, so that 5 chromatographs would be monitored instead of 20. Consulting the GC Manual (7), this appeared to be a relatively easy task to undertake. However, trying to apply this modification proved to be a problem. It was finally found that the choice of an odd number of chromatographs lead to difficulties in the program. Thus the maximum number of monitored chromatographs was changed to 6. Appendix A.2 gives specific detail of this modification.

5.3 Allocation of Inskel Common Area

It was decided to allocate a part of Inskel Common for other user's programs. The Fortran written GC programs originally started at the beginning of Inskel Common. Therefore, in order to allow the user 250 words at the start of Inskel Common, a dummy array IDUM(250) had to be inserted in all the GC Fortran programs. Consideration had to be given to the Assembler language programs, which use direct addresses of the variables in Inskel Common. Changes were made so that the starting address of Inskel Common was now specified in the Assembler programs symbol table less 250 to allow for the dummy array. However, problems developed when this was tested, and it was found that some of the Assembler language programs used the contents of a fixed core address, which contained the original starting address of Inskel Common +1. The changes that were made

are shown in Appendix A.3.

5.4 Combination of The GC, DDC, PØC Systems

When the Gas Chromatograph, Direct Digital Control and the Process Operators Console programs were combined, it was found necessary to specify a system skeleton equal to 26,000 words, leaving 7,000 words available for variable core. Originally, the GC program occupied five programmed interrupt levels. However, with the combined system, it was necessary to use only one interrupt coreload level and combine three of the original interrupt coreloads on this one level. The other two interrupt coreloads became mainline programs. This combination of coreloads on one interrupt level produced an interrupt coreload too large for the size of variable core in the computer. A solution was found by localizing the three programs and making a small interrupt coreload. This has the effect of bringing in the required program when called, rather than all three programs simultaneously. Specific details can be seen in Appendix A.4. With the combined system, a problem occurred with the chromatograph analog inputs. The system would cycle in a program, waiting for an operation complete flag to be set. This happened when an interrupt, operating on the same level as the analog input program (timer level), occurred during the actual analog input procedure, because the hardware analog input level was lower than the calling program. This was due to extra programs being operated via the timer interrupt, such as DDC. Previously with the dedicated GC system the timer interrupt was synchronized so that it never occurred when an analog input point was being brought into the computer. To avoid this problem, the GC basic cycle programs were all changed to operate at levels below the hardware analog input level.

5.5 Supervisory Program

It was considered essential to provide a supervisory program for the combined GC, DDC, PØC system for several reasons. Firstly, the GC basic cycle programs operating via Timer A interrupt should only be executed when a chromatograph is running, and not on a system cold start. The supervisory program would have the capability of turning off timer A if no chromatograph analysis is being performed or pending. Secondly, to have the capability of repeated analysis runs (continuous chromatograph), a supervisory program is required which would check whether a chromatograph analysis is to be reinitiated.

A simplified overall flow diagram, outlining the role of the supervisory program, is shown in Figure 5.3. It was decided that this supervisory program would be initiated using one of the computer's programmed timers, which would interrupt every supervisory program cycle time. This cycle time is currently set at 60 seconds, but can be changed by the analyst from the model 1816 typewriter keyboard, only when no chromatograph analyses are being performed or pending. A more detailed description of the supervisory program is given in Appendix A.5, and a flow diagram included in the modified GC Manual (7).

5.6 Continuous Chromatograph

The original GC program had no capability to reinitiate chromatograph analyses for use in process control, primarily because only laboratory chromatographs were considered. In order to apply a process chromatograph to this system, the continuous or repeated chromatograph feature becomes essential. Having already discussed the supervisory program addition, it would seem logical to apply this feature to control the reinitiation of the chromatographs. This reinitiation would occur at

an analyst specified time after completion of an analysis run. The repeated analysis information would be entered into the computer at the same time as the analyst entry specifications, via the model 1816 typewriter keyboard. This information now requires storage in the computer so that upon reinitiation the computer has the required data. Analyst entry information can be seen in Appendix A.1. More specific detail about the repeated analysis technique is outlined in Appendix A.6.

5.7 Capability to Change Analysis Methods For A Continuous Chromatograph

This modification will allow the analyst to perform a maximum of three different analyses with the same chromatograph, on a cyclic basis. This would be useful for example, when the analysis of a reactor input and output stream is desired using the same chromatograph. The modification is made in the program DCODE (consult the GC Manual (7) for the flow diagram).

Consulting Appendix A.1, it can be seen that the code numbers for the three methods, interpreted by the computer to analyze the three different streams, are entered in columns 13, 14, and 15 of the analyst first entry. If only two streams are to be continuously analyzed, then column 15 is left blank. This technique would allow a chromatograph to be run continuously with a standardization run being performed regularly, to maintain accurate standardization factors.

5.8 Off-line Processing of Chromatograph Raw Data

This technique was discussed in Chapter 2, whereby the raw chromatograph detector output data could be stored directly in a file on disk for later processing. Using this technique, it is assumed that the file data, analog-digital converter counts, is created by another program, such as a high speed data acquisition program. It was also considered

that this technique could only be used without any actual chromatograph operating, so that the sampling rate could be increased to a value greater than 5 points per second. A flow diagram of the routines involved in this technique is shown in Figure 5.2.

The author's programs have the capability to read the raw data into the file, providing the raw data is in punched card form. The cards are loaded into the computer via the card reader by pressing the console interrupt button with data switch nine on. The sampling rate can be changed from the keyboard and changes back to the original 5 points per second at the completion of a run. A disk read program is executed to transfer a sector of the file data into a core buffer. At chromatograph start up, the analog input is read from the core buffer and not from the analog-digital converter. The disk read program is called whenever the buffer is empty, to transfer the next disk sector of data to core for continued processing.

The applications of the combined GC, DDC, PØC system will be discussed separately in Chapter 6, since this is considered to be where the main advantages of the combined system appear. A simplified overall flow diagram and a detailed overall flow diagram of the modified system, shown in Figures 5.3 and 5.4 can be compared directly with the original IBM flow diagrams, Figures 4.1 and 4.2 respectively in Chapter 4.

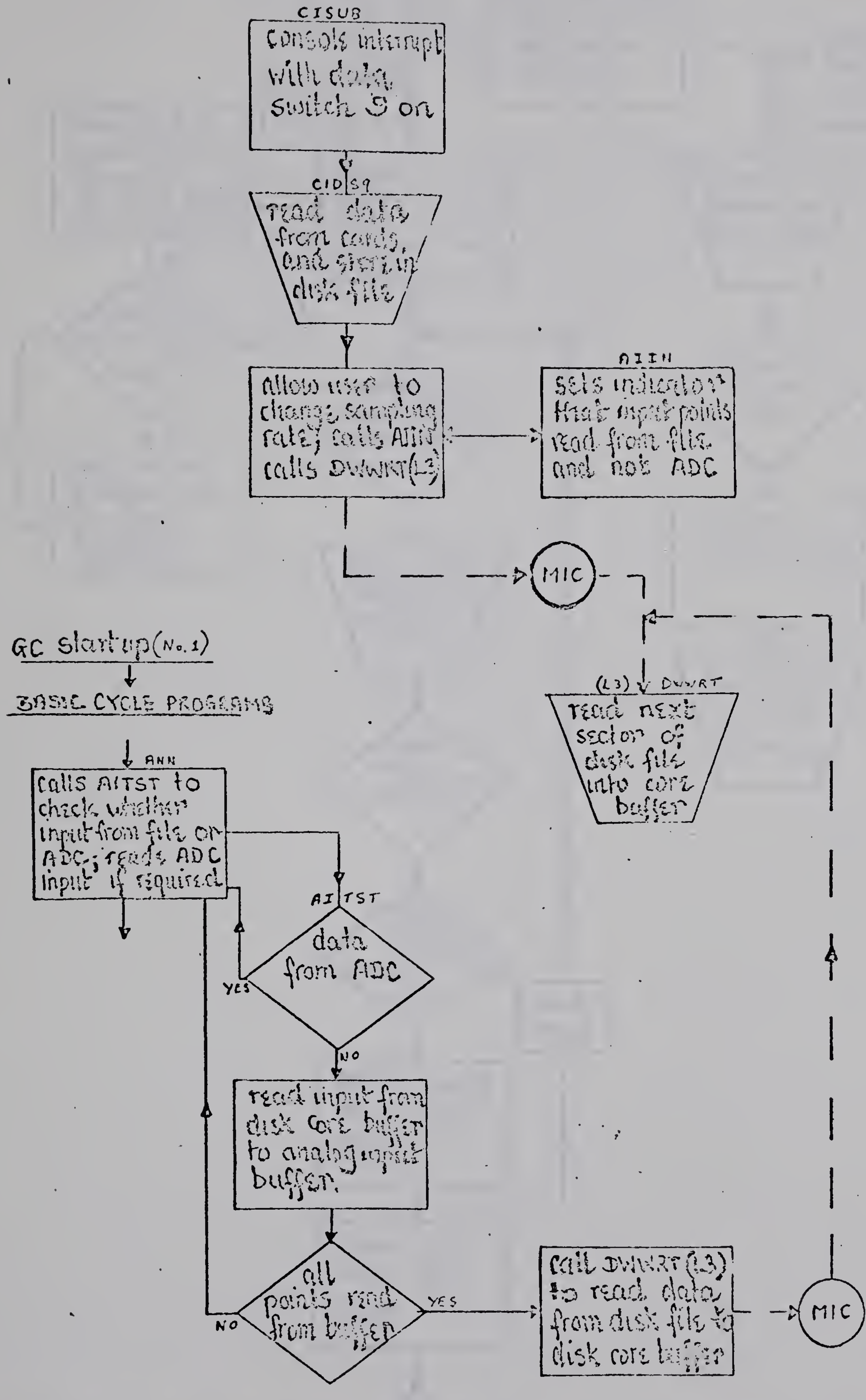


Figure 5.2 Overall Flow Diagram of the Off-line Processing Technique

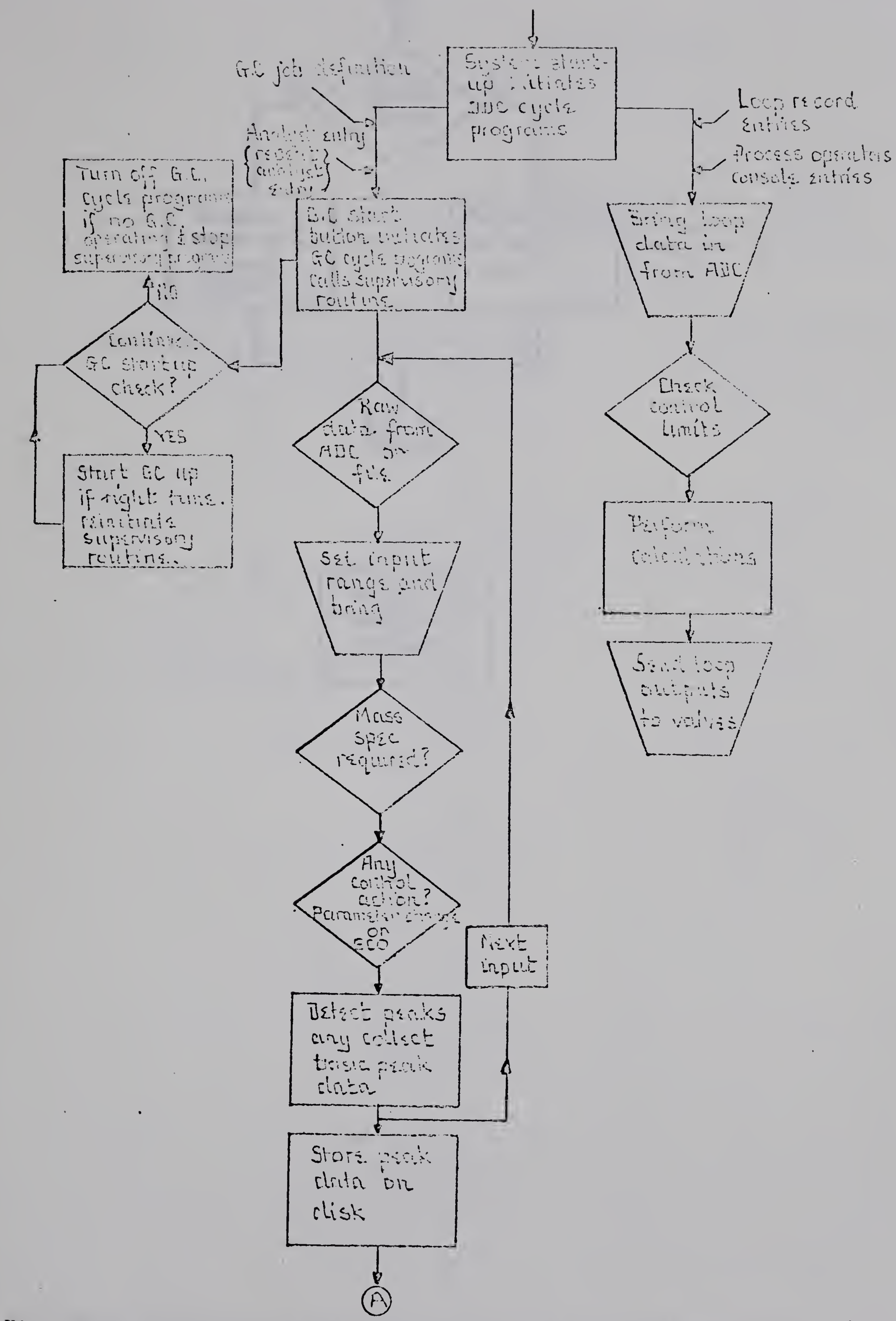
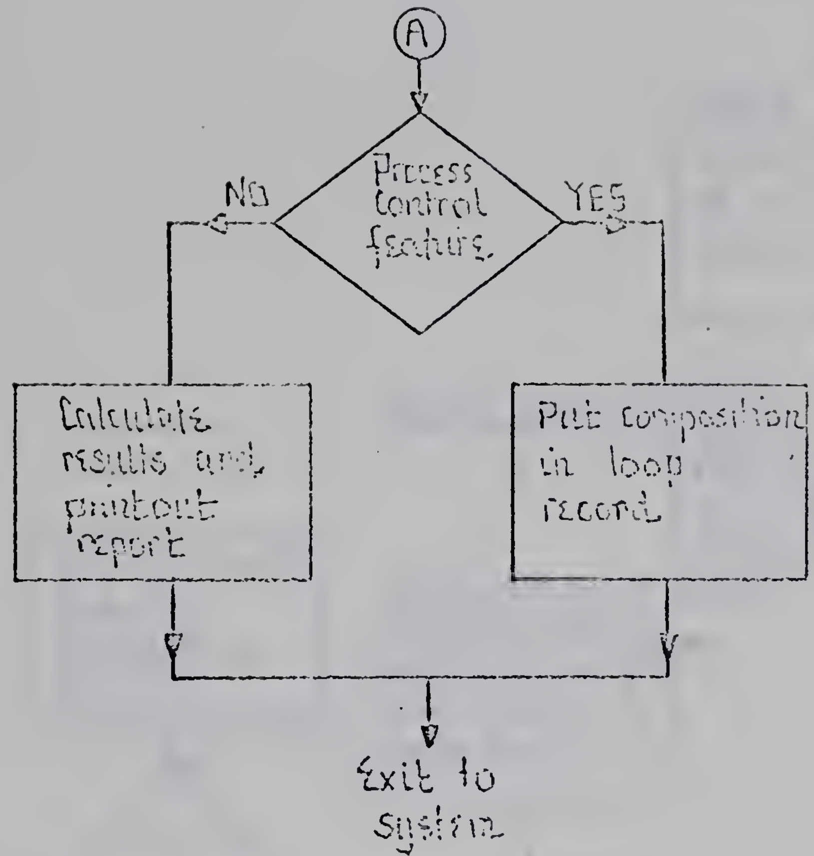
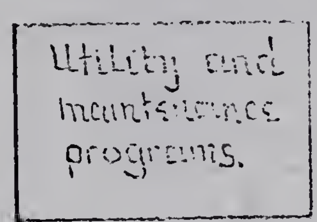


Figure 5.3 Simplified Overall Flow Diagram of the Modified System


A
Process control feature
NO
YES
Calculate results and printout report
Put composition in loop record
Exit to system


Utility and maintenance programs.

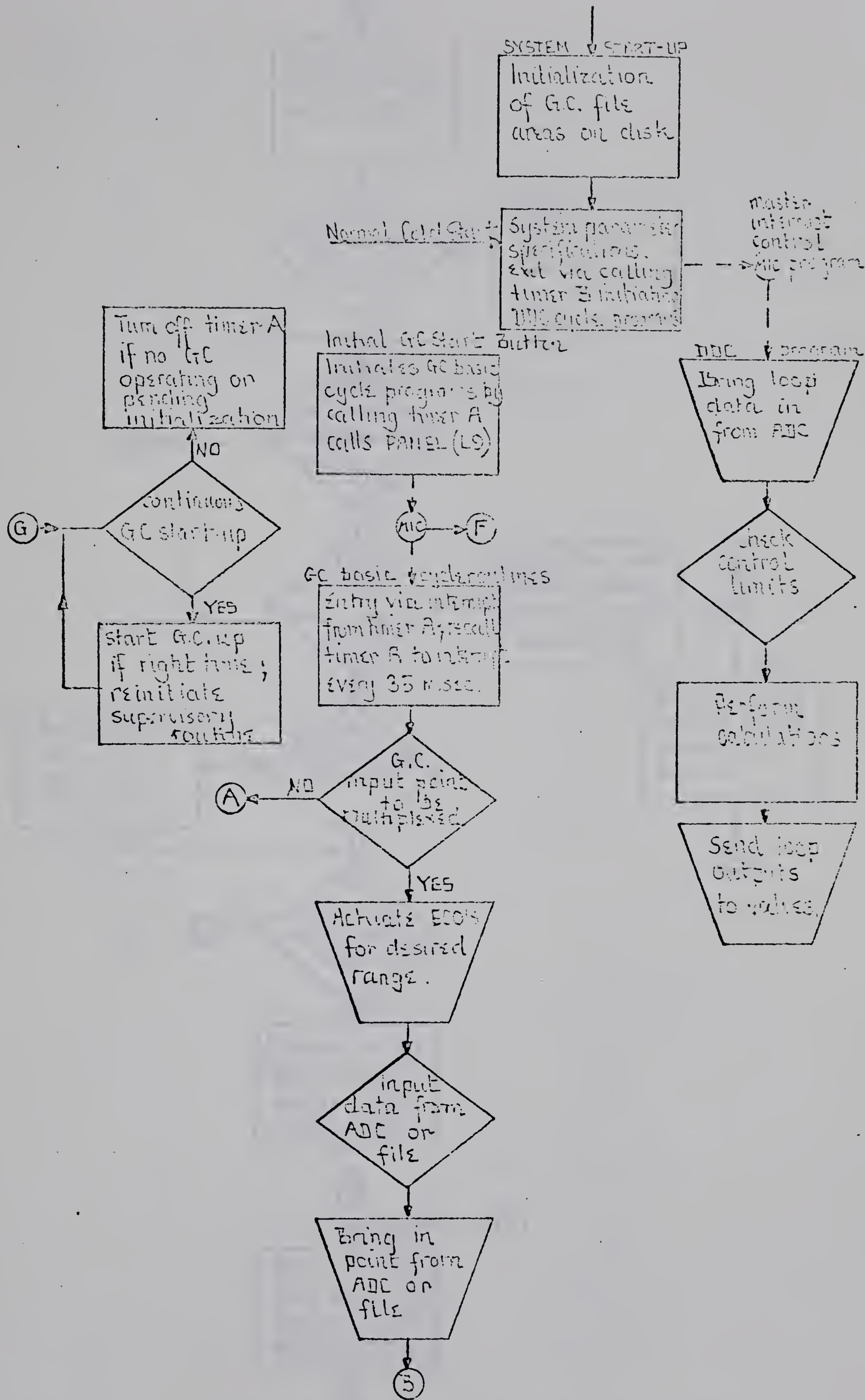
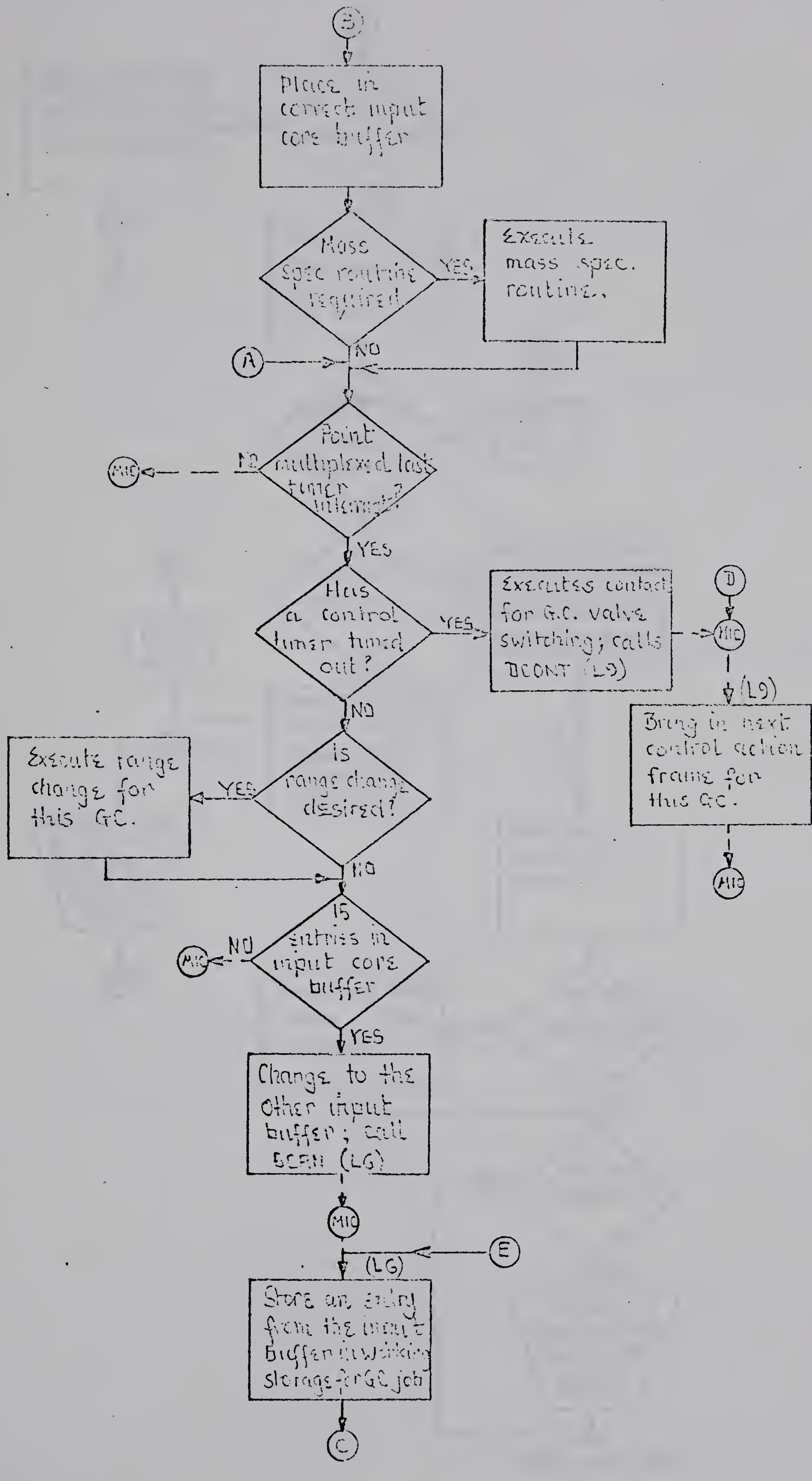
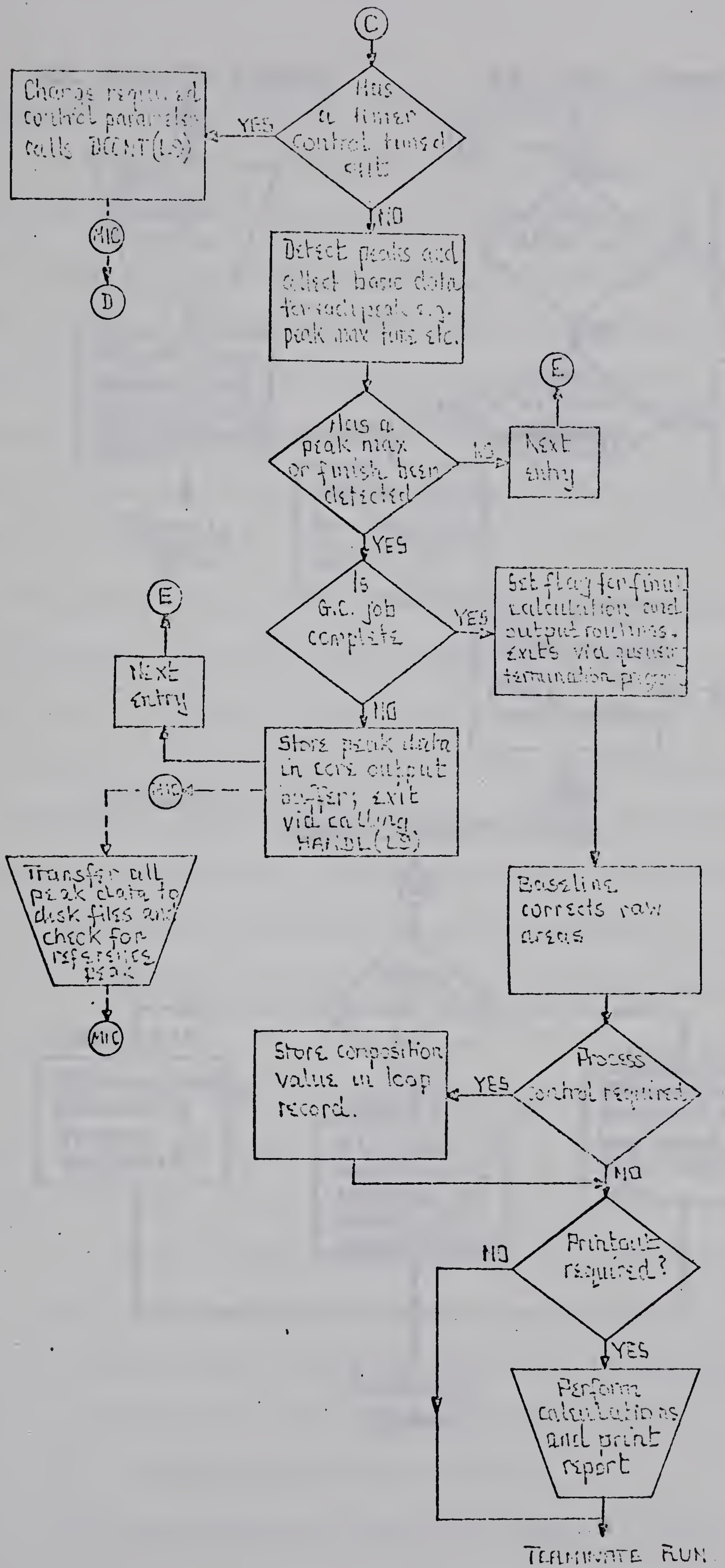
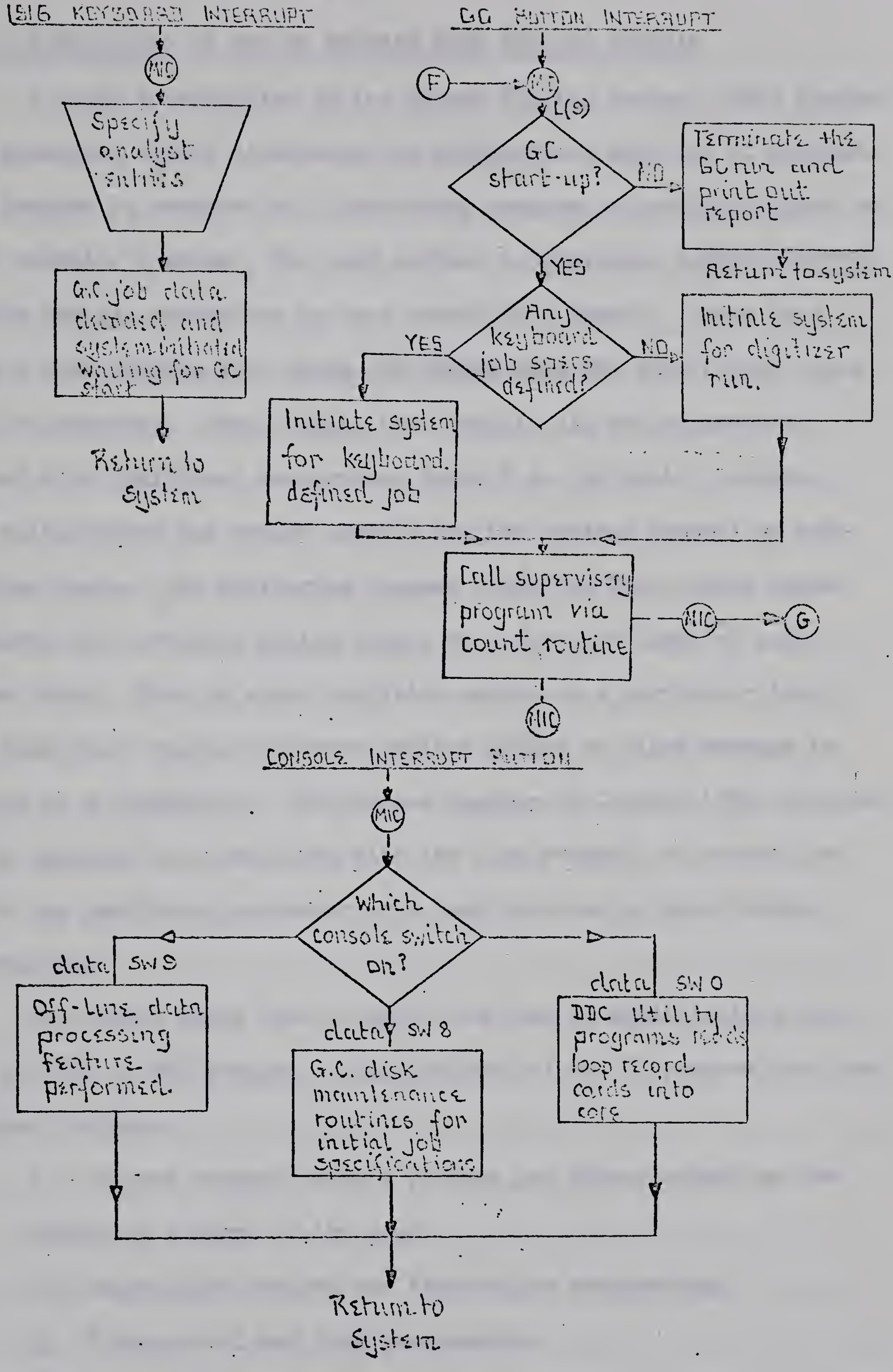


Figure 5.4 Overall Flow Diagram of the Modified System


B
Place in correct input core buffer
Mass spec routine required
YES
Execute mass spec. routine.
NO
A
Point multiplexed last timer interrupt?
NO
MIC
YES
Has a control timer timed out?
YES
Executes contact for G.C. valve switching; calls DCONT (L9)
D
MIC
(L9)
Bring in next control action frame for this GC.
MIC
NO
Is range change desired?
YES
Execute range change for this GC.
NO
Is entries in input core buffer
NO
MIC
YES
Change to the other input buffer; call SCAN (L6)
MIC
E
(L6)
Store an entry from the input buffer in working storage for GC job
C


C
Has a timer control timed out
YES
NO
Detect peaks and collect basic data for each peak e.g. peak max time etc.
Has a peak max or finish been detected
Next entry
E
YES
Is G.C. job complete
YES
Set flag for final calculation and output routines. Exits via queuing termination program
NO
Store peak data in core output buffer; exit via calling HANDL(LS)
Next entry
E
MIC
Transfer all peak data to disk files and check for reference peak
MIC
Baseline corrects raw areas
Process control required
YES
Store composition value in loop record.
NO
Printout required?
NO
YES
Perform calculations and print report
TERMINATE RUN
MIC
D


1816 KEYBOARD INTERRUPT
MIC
Specify analyst entries
G.C. job data decoded and system initiated waiting for GC start
Return to System
GC BUTTON INTERRUPT
F
MIC
L(9)
GC start-up?
NO
Terminate the GC run and print out report
Return to system
YES
Any keyboard job specs defined?
YES
NO
Initiate system for keyboard defined job
Initiate system for digitizer run.
Call supervisory program via count routine
MIC
G
MIC
CONSOLE INTERRUPT BUTTON
MIC
Which console switch on?
data SW 9
Off-line data processing feature performed.
data SW 8
G.C. disk maintenance routines for initial job specifications
data SW 0
DDC Utility programs reads loop record cards into core
Return to System

6. APPLICATION OF THE GC PROGRAM WITH THE DDC PROGRAM

A brief introduction of the Direct Digital Control (DDC) program will be presented before discussing its applications with the GC program. The DDC program is composed of a monitoring program, a loading program and an error checking program. The user defines a particular control loop by specifying certain parameters on loop record data sheets. These data sheets are then punched into cards and loaded into the loop record table area of the computer. These tables then contain all the parameters associated with individual measurement (word 8 in the table), alarms, control calculations and output signals for the various control or data acquisition loops. The monitoring program scans the loop record tables and performs the necessary analog inputs etc. for each loop, at user specified times. When an alarm condition exists on a particular loop, such as high input value, the error routine causes an alarm message to be printed on a typewriter. The Process Operator's Console (PØC) routines allow the operator to communicate with the loop records, to obtain the status of any particular parameter in a loop and also to make changes where required.

The author would like to point out that no modifications were made to the DDC or PØC programs. Applications of the GC program with the DDC program include:-

1) Process control using a process gas chromatograph as the detecting element of the loop.
2) Temperature control and temperature programming.
3) Flow control and flow programming.
4) Chromatograph protection features.
5) Chromatograph monitoring with the DDC program.

6.1 Process Control

If process control of some composition loop is desired using a process gas chromatograph as the detecting element of the loop, the repeated analysis feature, discussed in Chapter 5.6, would have to be specified in the analyst keyboard entries. Normally for control purposes, one major component in the sample would suffice as the measured variable. This would imply defining a job with only this one peak to be detected, and once detected the analysis would be complete. The analyst has the option of obtaining control alone or control with printout for this one peak. The programming modifications to allow complete printout of all the peaks combined with the control feature was not undertaken by the author, although this would appear a useful technique. Using the control option, the measured composition value is transferred to the correct DDC loop record table, specified in the analyst keyboard entries. This program only has the capability to use one component for control purposes, so the job definition must be specified to detect this one peak alone.

The analyst keyboard entries, as shown in Appendix A.1, will now be specified for this type of analysis. A DDC loop record identification number must be entered in column 10 of the first analyst entry so that the computer can store the measured composition in the desired loop record. A continuous chromatograph analysis must be specified to the computer with or without the printout option. This is performed by typing '1' in column 12 of the first analyst entry for control without printout, '2' for control with printout and '3' for a continuous chromatograph analysis with printout. Column 11 must contain an entry to specify the delay, before reinitiation of the chromatograph is desired.

One problem that had to be given careful consideration was the

storing of inaccurate composition values in the loop records. This is partially taken care of by the DDC program, which would inform the chromatograph analyst of the error, by causing an error printout stating that the value cannot be changed for some reason, such as value too high or loop record not valid. An example of the analyst entries and results obtained for this control technique is shown in Appendix A.15.

6.2 Temperature Control and Temperature Programming

Since temperature control of the chromatograph analyzer compartment is a necessary feature, to enable true comparison of the results for similar samples and methods used, the DDC program could handle this as a separate temperature control loop. In order to have the capability of programmed temperature control throughout a chromatograph analysis, there has to be a transfer of data between the two major programs. For example, during an analysis run, the chromatograph program must provide new set point values to a particular temperature loop record. It would be advantageous, if by transferring two values to the loop record, such as the new set point value and the time requested for the loop output to reach this value, the DDC program had the capability to increase the temperature loop output at the desired rate. Since the DDC program at the University of Alberta does not have this capability, which has been applied in industry, one method which would solve the task is as follows.

The GC program has the capability to change control parameters, such as smoothing factors, at any time during a chromatograph analysis. These preprogrammed changes in the control parameters are specified for the particular analysis method via punched cards at job definition time. The existing programs were modified and new programs written to apply this technique to the system. However, the inclusion of the new programs and

communication programs created extra requirements on the size of the system skeleton which amounted to exceeding the specified 26,000 words. For this reason, the technique could not be applied, but the documentation and program listing with the required modifications have been included in the modified system documentation manual (7). If these modifications could have been made, the temperature loop set point and output change limit could have been changed at any time throughout an analysis run. This would then have allowed the temperature programming feature to be achieved, since the cycle time of the temperature loop would be known.

A simplified example, shown in Figure 6.1, using this technique shows the required changes in set point and output change limit for a particular temperature programmed analysis. Assuming the cycle time of the temperature loop to be five seconds, then the required changes in set point and output change limit at time T1 are;

new set point = 150°F

new output change limit = 5°F

This would allow a change of 5°F to be made over a period of five seconds. This technique assumes that the chromatograph hardware is capable of implementing these changes.

6.3 Flow Control and Flow Programming

The flow control and flow programming features of the chromatograph carrier gas could be implemented by the DDC program in a manner similar to the corresponding temperature features, discussed in section 6.2.

6.4 Chromatograph Protection Features

The DDC program has the capability to monitor the chromatographs for critical conditions, such as excessive detector filament current or low air flow rate over the heater elements, previously discussed in Chapter

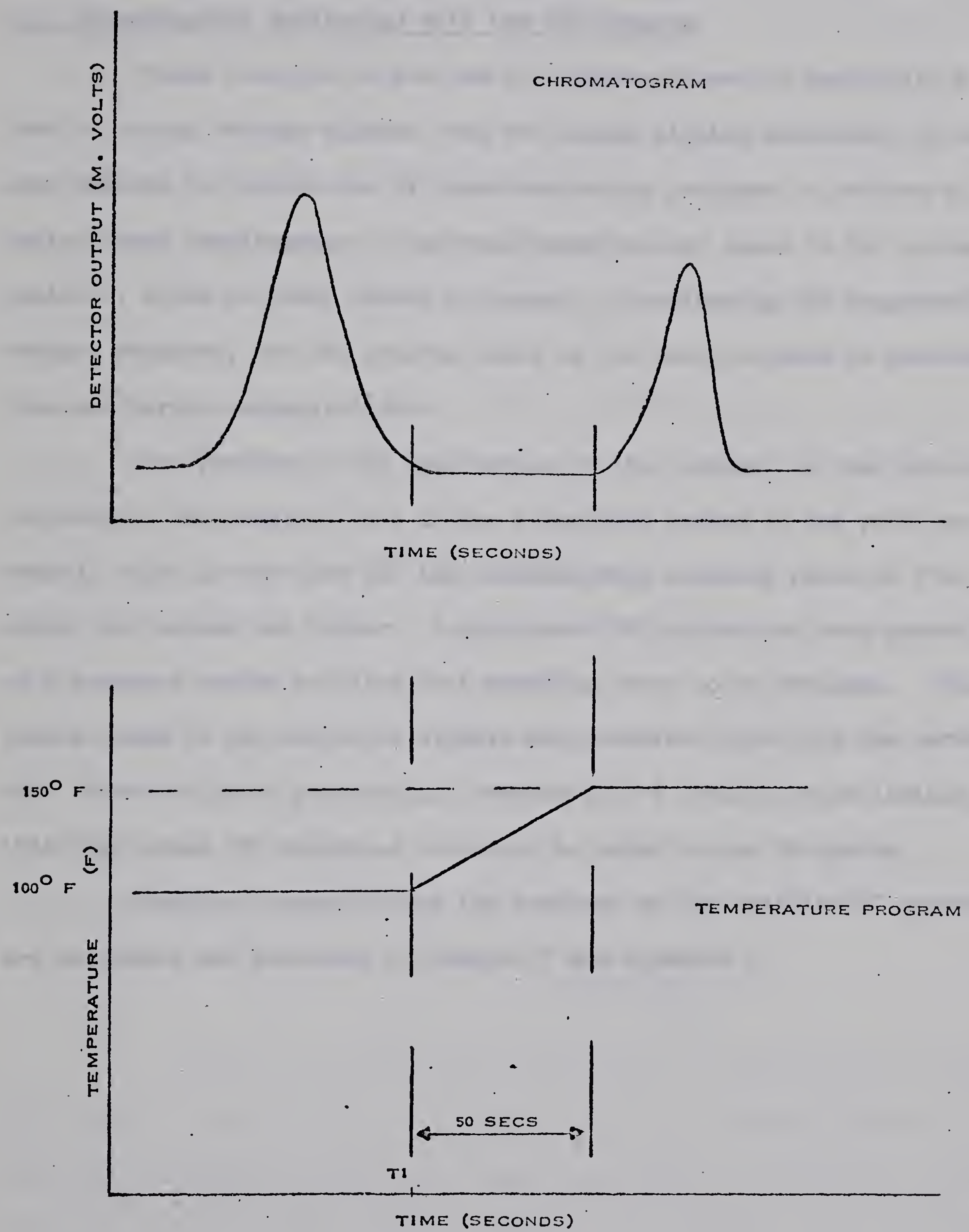


Figure 6.1 Temperature Programming Diagram

2. The computer could inform the operator of an alarm condition by an error printout, and could even shut the chromatograph power off in an emergency condition by means of its external contact points.

6.5 Chromatograph Monitoring with the DDC Program

Since both the GC and the DDC programs have the capability to read in analog voltage signals from the analog digital converter, it would seem logical to utilize one of these monitoring programs to perform all the analog input requirements. This would save storage space in the system skeleton, which is badly needed at present. Considering the programming changes required, the DDC program would be the easier system to undertake this monitoring responsibility.

One problem in the application of this change, is that presently the maximum DDC sampling rate in the integrated system is one point per second, which is too slow for the chromatograph sampling rates of five points per second and higher. A high speed DDC system has been generated as a separate system to allow fast sampling rates to be obtained. This system reads in the raw input signals and transfers them into the correct disk files for later processing. Because of the skeleton size limitation, this high speed DDC technique could not be added to the GC system.

Results demonstrating the features of the modified GC program are presented and discussed in Chapter 7 and Appendix C.

7. DEMONSTRATION OF THE FEATURES OF THE GC PROGRAM WITH DISCUSSION OF RESULTS

The following chapter and referenced appendix are presented in a self-sufficient manner, so that they might serve as the operating procedures manual for the chromatograph analyst. Before considering the computer-chromatograph system, an outline of the manual-chromatograph analyst procedures will be discussed.

7.1 Manual-Chromatograph Analyst Procedures

Assuming that the chromatograph operating conditions, such as column temperature, have been attained, the analyst would inject a standard sample of a mixture of components into the chromatograph. This would result in a chromatogram being traced out on the attached recorder, an example being shown in Figure 7.1. The chromatogram is then used by the analyst to calculate standardization factors for each peak, such as peak A and peak B in Figure 7.1. These factors can then be applied in the analysis calculations of an unknown sample of a similar mixture. If the analyst applies baseline correction to the peak area calculations, then the two peak areas shown in Figure 7.1 would be calculated. Since the peak area A is a function of the concentration of A in the mixture, a standardization factor for peak A can be determined as follows:

$$(\text{standardization factor})_A = \frac{(\text{concentration})_A}{(\text{Peak area})_A}$$

A similar calculation will determine the standardization factor for peak B. Having acquired these factors using a standard sample of a mixture, an unknown analysis can then be undertaken to obtain the concentrations of A and B in an unknown sample of the mixture. The concentrations are calculated by determining the peak areas A and B from the unknown

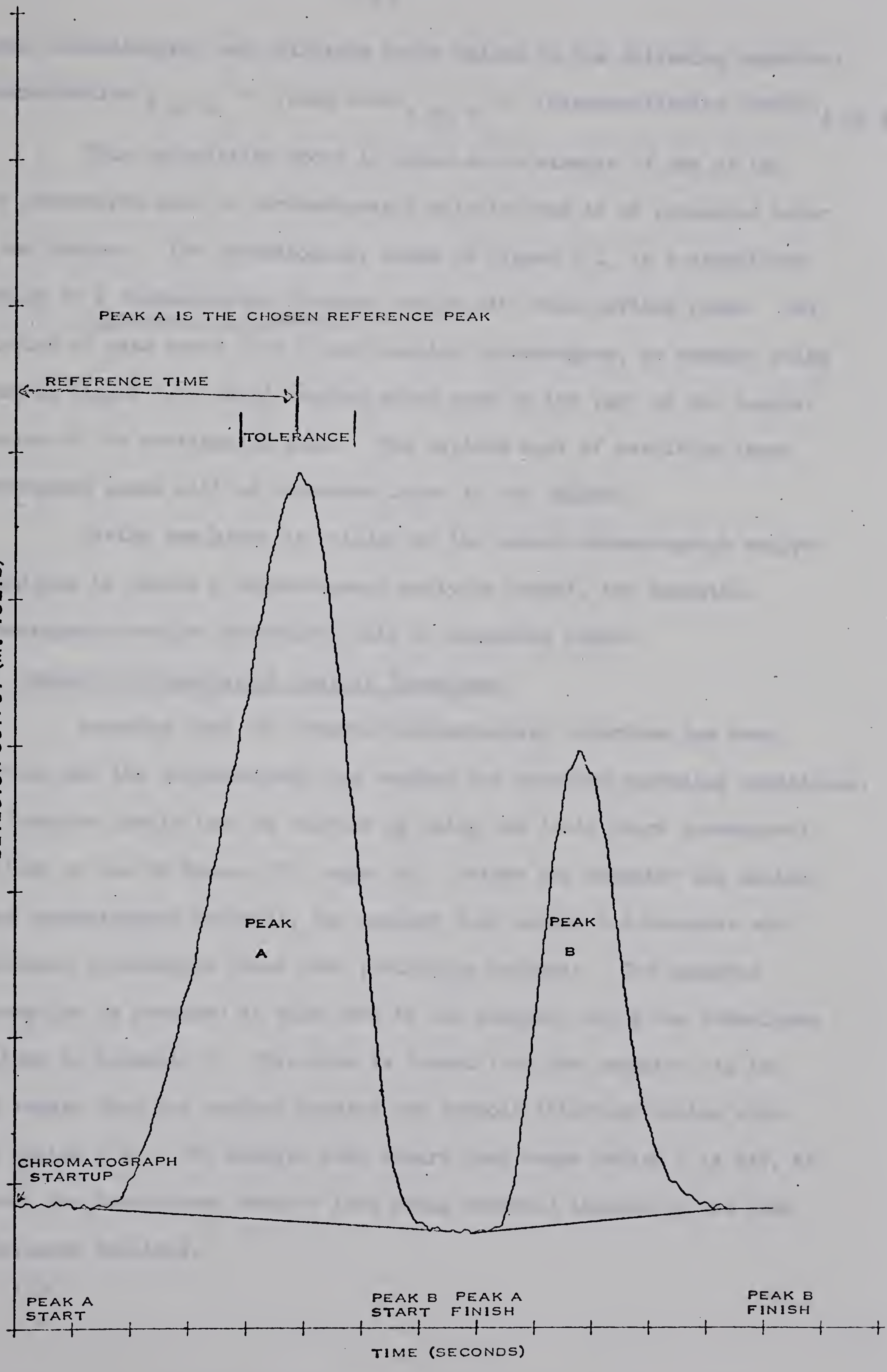


Figure 7.1 Chromatogram Number 1

sample chromatogram, and utilizing these values in the following equation:

$$(\text{concentration})_{A \text{ or } B} = (\text{peak area})_{A \text{ or } B} * (\text{standardization factor})_{A \text{ or } B}$$

This calculation above is given as an example of one of the many procedures used in chromatography calculations to be presented later in the chapter. The chromatogram, shown in Figure 7.1, is a simplified version of a chromatograph detector output with well defined peaks. Calculation of peak areas from a more complex chromatogram, an example being shown in Figure 7.2, would require extra care on the part of the analyst because of the overlapping peaks. The various ways of resolving these overlapping peaks will be discussed later in the chapter.

Having now given an outline of the manual-chromatograph analyst techniques to obtain a chromatograph analysis report, the computer-chromatograph analyst procedures will be presented below.

7.2 Computer-Chromatograph Analyst Procedures

Assuming that the computer-chromatograph interface has been provided and the chromatograph has reached the required operating conditions, the computer should then be started up using the 'cold start procedures', outlined in the GC Manual (7), page 240. Before the computer can monitor a gas chromatograph analysis, the analyst must supply the computer with sufficient information about that particular analysis. The analysis information is produced in card form by the analyst, using the techniques outlined in Appendix B. This data is loaded into the computer via the card reader when the analyst presses the console interrupt button with data switch 8 on. The analyst must ensure that sense switch 7 is off, to prevent the Non-Process Monitor from being executed instead of the Disk Maintenance Routines.

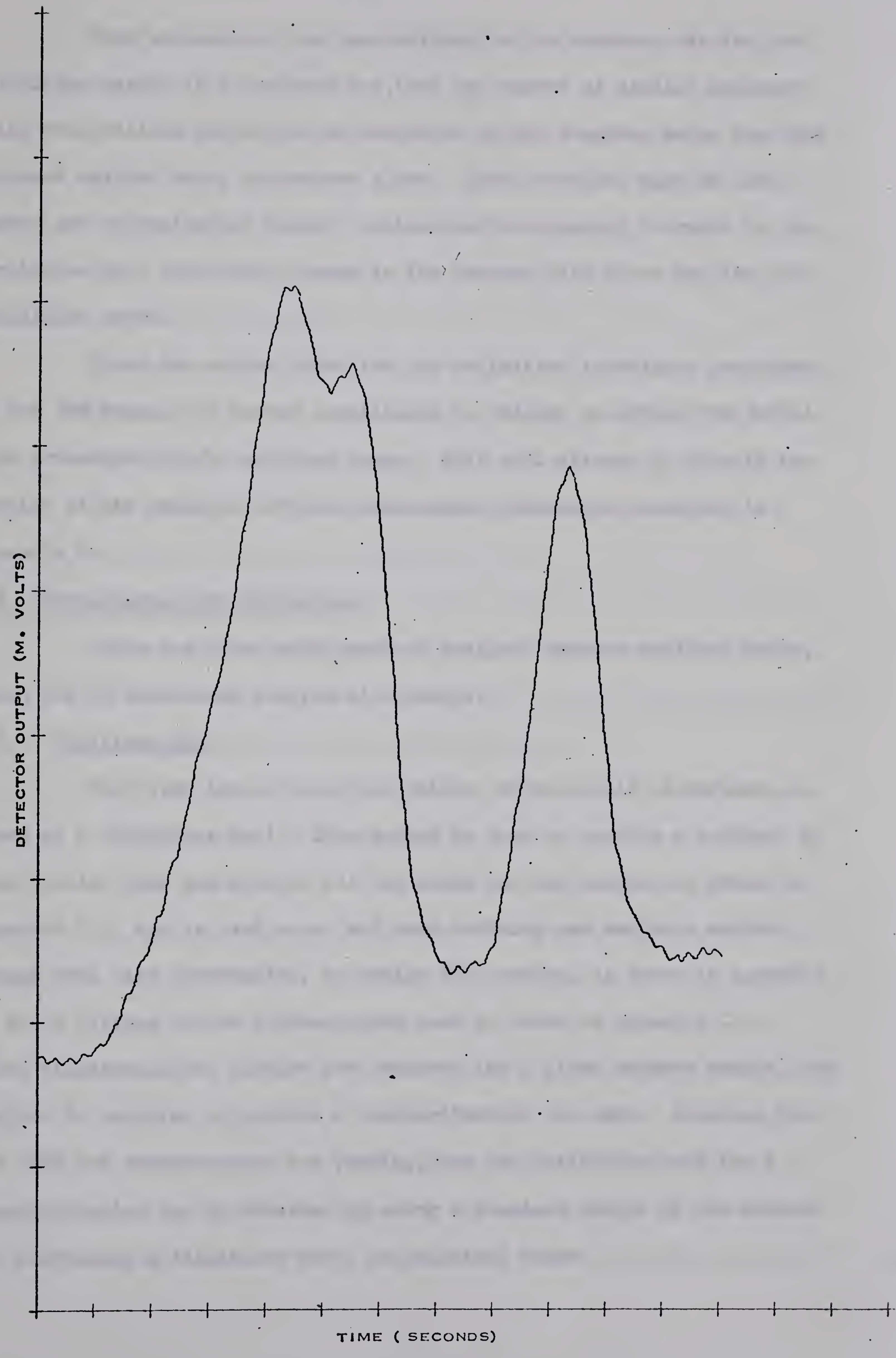
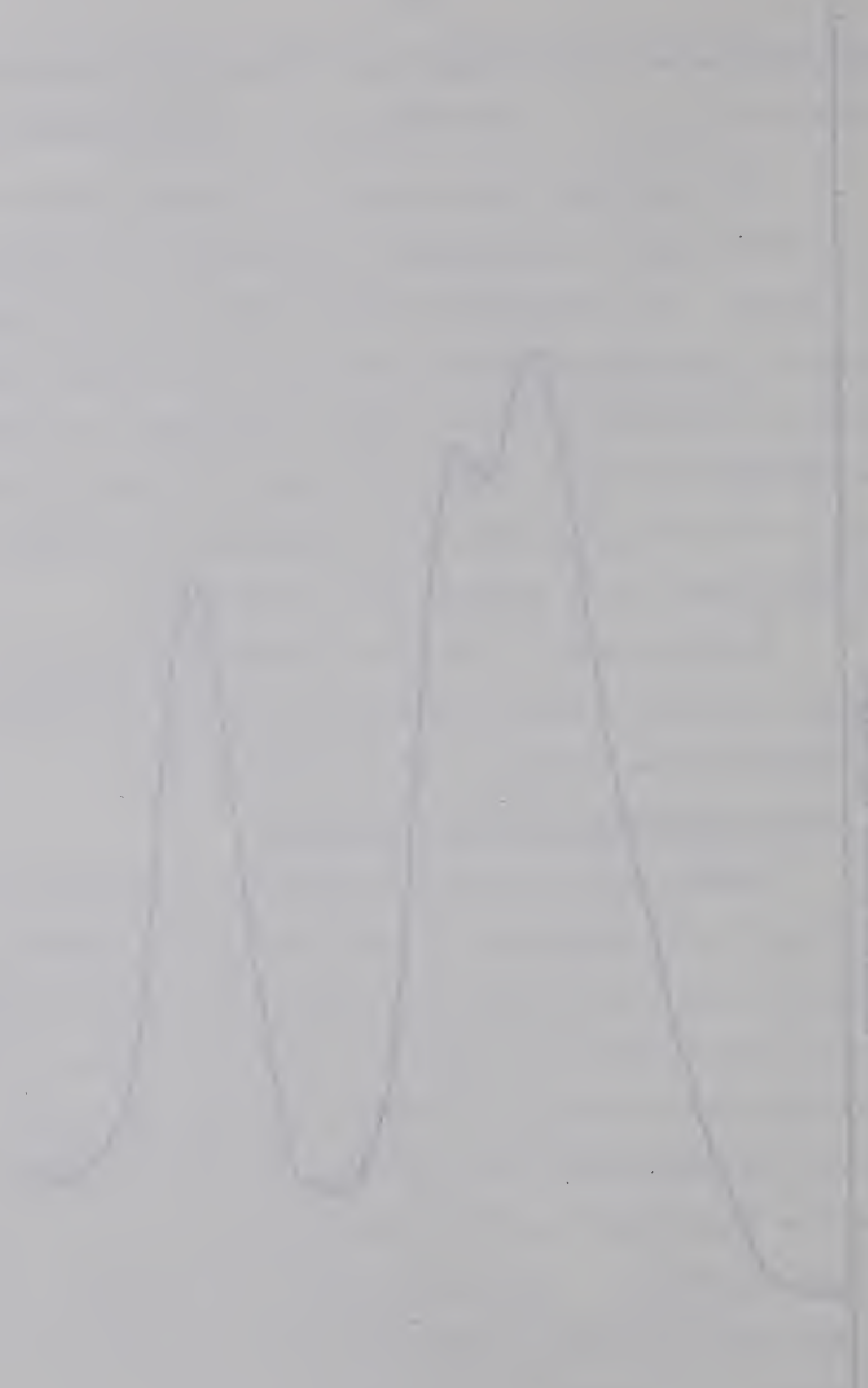


Figure 7.2 Chromatogram Number 2



Once an analysis has been defined to the computer via the job definition cards, it is pointed out that any number of similar analyses using this defined method can be monitored by the computer using the 1816 keyboard analyst entry procedures alone. These entries, such as job number and chromatograph number, initialize the computer to refer to the particular job, previously stored in the correct disk files via the job definition cards.

Since the author found the job definition techniques presented in the IBM Manual (7) rather complicated to follow, an actual job definition procedure will be outlined below. This will attempt to clarify the reading of the modified IBM Disk Maintenance Procedures presented in Appendix B.

7.3 Chromatograph Job Definition

There are three main types of analysis methods outlined below, which the GC monitoring program will handle.

7.3.1 Digitizer Run:

The first type of analysis method, which should be defined, is known as a 'digitizer run'. This method is used to provide a printout of peak elution time and area of all the peaks for any sample, as shown in Appendix C.2, and is used as an aid when defining new analysis methods. Actual data card information, to define this method, is given in Appendix B, and a listing of the punched cards used is shown in Appendix C.2. Since standardization factors are required for a given unknown sample, the analyst is required to perform a standardization run next. Assuming that the DACS and chromatograph are running, the job definition data for a standardization run is obtained by using a standard sample of the mixture and performing a 'digitizer run', as explained below:

1) Press the chromatograph button and wait until the sample inject light comes on.

2) Inject the standard sample of the mixture into the chromatograph. This is usually done by syringe if the sample is liquid, or by a sample valve if the mixture is gaseous. These two steps are sufficient to indicate to the computer that a 'digitizer run' is to be carried out.

3) When the chromatograph has traced out all the required peaks on a recorder with known time base on the X-axis, similar to Figure 7.1, the chromatograph button should be pressed again. This signals the computer to terminate the job and produce a printout, as shown in Appendix C.2.

4) Using the chromatogram and the 'digitizer run' printout as a time check, the following information can then be determined:

a) one of the peaks, preferably a large, well defined peak, should be chosen as a reference peak.

b) the reference time, ie. the elution time of this reference peak in seconds is taken.

c) a tolerance for the reference time is chosen, consideration being given to how much the reference peak might be displaced along the time axis without another peak being mistaken as the reference peak.

d) the finish time for the chromatograph analysis in seconds is chosen.

e) the peaks for each component are given time bands in which they are to occur on the chromatogram, as shown in Figure 7.1, ie. the earliest time for a peak start and the

latest time for a peak finish. The user can specify overlapping time bands, shown for the two peaks in Figure 7.1, but care must be taken so that a peak maximum is not detected in the wrong time band.

f) control action times are also specified. For example, times are specified to operate external contacts for back-flushing or change the various control parameters, such as smoothing factors. The external contact times, defined in the job definition, can either be specified relative to the reference peak elution time or as absolute values. The specified times for control parameter changes can only be defined relative to the reference peak elution time.

7.3.2 Standardization Run:

Once the data from the digitizer run has been obtained, the job definition cards defining the actual standardization run can then be punched out, as specified in Appendix B.1. Two techniques can be used for standardization runs as indicated below:

1) external run: the factors obtained from the run are printed out on a report, as shown in Appendix C.9 and C.10, which can then be used by the analyst to fill out the unknown job definition cards.

2) internal run: when this technique is employed, the factors obtained are automatically stored in the desired unknown job method as well as being printed out in a report, as shown in Appendix C.13. This saves the analyst the need to enter the factors on the unknown job definition cards. It is important to note however, that the unknown job must already be stored in

the correct disk files, previous to the internal standardization run. This feature is useful to update the factors used in previously defined jobs.

An actual listing of the job definition cards for an external standardization run is shown in Appendix C.9. The standardization run, once defined, can then be carried out to obtain the correction factors for each peak. The analyst procedures are outlined below:

1) specify the desired job number and chromatograph number, etc., using the analyst entries via the model 1816 typewriter keyboard.

2) press the chromatograph button and wait for the start up light before sample injection. The computer will then determine the factors and terminate the run automatically.

7.3.3 Unknown Run:

The unknown job should now be defined, as specified in Appendix B.2, since the analyst now has the required peak standardization factors for the unknown samples. Once this job has been defined to the computer, the analyst operating procedures for an unknown run are as follows:

1) specify the desired job number and chromatograph number, etc. using the analyst entries via the model 1816 keyboard.

2) press the chromatograph button and wait for the start up light before sample injection. The computer will then calculate and print out the results, after the computer automatically terminates the analysis run.

An actual listing of the job definition cards for an unknown run is shown in Appendix C.4.

There are various options, described in Appendix B, which the analyst can use when defining a job with regard to peak area calculations

and final report output calculations. Several examples using these various options, showing job definition, analyst 1816 keyboard entries and results, are included in Appendix C. These examples also include jobs using the author's modifications made to the original chromatograph program, discussed in Chapter 5. This chapter will now continue with a discussion of the results obtained, in an attempt to demonstrate the features of the GC monitoring program.

7.4 Discussion of the Results

This section will discuss each of the chromatograph job examples, shown in Appendix C, separately to explain the various techniques available with the GC program.

7.4.1 Reference Peak Selection

The choice of a reference peak required in the job definition procedures, discussed in Chapter 5, should make no difference to the final results with the exception that a shoulder peak must not be selected, according to the documentation in the original IBM GC manual (7). If any chromatograph operating variable changes slightly, such as carrier gas flow rate, the elution time of the peaks will be displaced by a certain amount. Because of this, the reference peak elution time should have as large a tolerance as possible without another peak being mistaken as the reference peak. Therefore, a well defined separate peak would be the best choice. Jobs 50 and 51, as shown in Appendix C, show the results of choosing two different reference peaks for the same computer data input. No significant difference was found in the results, shown in Table 7.1, which therefore verifies the above discussion.

7.4.2 Peak Area Calculation Options

The options provided in the GC program for peak area calculations,

TABLE 7.1

SUMMARY OF THE CHROMATOGRAPH JOB RESULTS

CHROMATOGRAM NO. 1

Component	JOB NUMBER 20	23	24
BENZENE	23.947	28.780	24.254
TOLUENE	8.683	10.442	22.291
ALCOHOL	14.716	17.115	14.566
WATER	35.321	39.989	35.495
GLYCOL	17.330	3.672	3.392

CHROMATOGRAM NO. 2

Component	JOB NUMBER 50	51	52	55	56	57
PROPANE	22.491	22.986	27.165	0.1709E- 2	0.1000E 1	0.600
BUTANE	27.180	27.128	13.656	0.1460E- 2	0.8215E 0	0.723
PENTANE	50.327	49.884	59.178	0.8060E- 3	0.4494E 0	1.293

	58	59	60
PROPANE	23.123	0.9999E 0	99.999
BUTANE	27.161	0.8418E 0	100.158
PENTANE	49.715	0.4567E 0	108.565

outlined in Appendix B, are demonstrated by the results of Jobs 20, 50, 52, 23 and 24 as shown in Appendix C and Table 7.1. Jobs 20 and 50 use the same option for all the peaks ie. with JTYPE specified as one for every peak. Job 52 is defined with the option JTYPE specified as two for the second peak and one for the remaining peaks. Comparing the results of Jobs 50 and 52 in Table 7.1 demonstrates clearly the difference in the value of the second component, which also affects the values of the other components since the results are normalized. Job 23 is defined with the fifth peak, a shoulder peak shown in Figure C.2, having an area calculation option JTYPE specified as three while the remaining peaks use option one. Comparing the results of Jobs 20 and 23 in Table 7.1 clearly indicates the difference in the area calculated for this peak. Job 24 is defined similarly to Job 23 except that peak number five uses peak area calculation option four. Using peak option four, the user has to specify one of the remaining peaks to accept the unwanted additional peak five area. Peak two has been specified in Job 24 to accept this extra area. The results of Jobs 20, 23 and 24, shown in Table 7.1, demonstrate the effect of these peak area calculation options using the same standardization factors for each job.

7.4.3 <u>Final Report Calculation Options</u>

The seven report calculations provided in the GC program, discussed in Appendix B, are demonstrated with Job examples 50, 55, 56, 57, 58, 59 and 60, shown in Appendix C. Job 55 is defined as an external standardization run with the analytical calculation number KALC being specified as one. The standardization factors obtained for each of the peaks, shown in Table 7.1, are calculated using the following equation:

$$(\text{Standardization Factor})_{\text{Peak } k} = \frac{(\text{Known Concentration})_k}{(\text{Area})_k}$$

Job 56 is defined as an internal standardization run, KALC being specified as two. The standardization factors obtained in this run are shown in Table 7.1 and are also stored on disk in job file for use with a specified unknown job. The equation for this calculation is

$$(\text{Standardization Factor})_{k \text{ stored}} = \frac{(\text{Standardization Factor})_k}{(\text{Standardization Factor})_{\text{ref}}}$$

Therefore, relative standardization factors are calculated which tend to eliminate sample size variation problems. The results of the unknown job using these results must be normalized to obtain the correct results as shown with options 4 and 5 in Jobs 57 and 58.

The final report output option three is used in Job 57, shown in Appendix C. This technique is used in an unknown job when the peak standardization factors are known and normalization of the final results is not required. The equation for this calculation for peak k is shown below.

$$\text{Concentration}_k = \text{Area}_k * (\text{Standardization Factor})_k$$

Job 50, shown in Appendix C, is defined using option four output calculation. The results of this calculation excluding unknown peak results are normalized as shown by the following equation

$$\text{Norm. Conc.}_k = \frac{\text{Un-norm Conc.}_k}{\Sigma\,\text{Conc.}_i} * (\text{Coded Normalization Conc.})$$

Job 58, shown in Appendix C, is defined with option five output calculation. This is an unknown run similar to Job 50, except that any unknown peaks detected are given standardization factors of 1.0 and are

included in the normalization. Since no unknown peaks were detected in either Job 50 or 58, the results shown in Table 7.1 are approximately the same.

Job 59, shown in Appendix C, is defined as an internal standardization run with KALC specified as six. This job calculates factors and stores them on disk in a specified unknown job file for later use with the unknown job as well as providing a printout of the factors. The unknown job must be defined with a KALC equal to seven. The standardization factor for peak k is calculated using the following equation.

$$(\text{St. Factor})_k = \frac{\text{Known conc.}_k * \text{Area}_{REF} * \text{Known (St. Factor)}_{REF}}{\text{Known Conc.}_{REF} * \text{Area}_k}$$

Known conc._k = the known concentration of the component producing the peak in the standard sample.

Area_{REF} = the area of the reference peak.

Known $(\text{St. Factor})_{REF}$

= the user specified reference peak factor, which the remaining peak factors are calculated relative to.

Known Conc._{REF} = the concentration of the chosen reference peak.

Area_k = the area of peak k in the sample.

The above parameters refer to the standard sample. The factors obtained, shown in Table 7.1, were used with Job 60 below.

Job 60, defined with KALC equal to seven, is an unknown run using the internal standardization factors provided by Job 59. The concentration results shown in Table 7.1 are calculated using the following equation.

$$(\text{Conc.})_k = \frac{\text{Area}_k * \text{St. Factor}_k * \text{Known Conc.}_{REF}}{\text{Area}_{REF} * \text{Known (St. Factor)}_{REF}}$$

$Area_k$ = the area of peak k in the unknown sample.

$St.\ Factor_k$ = the standardization factor obtained from an internal standard run for peak k.

$Known\ Conc._{REF}$ = the user specified reference peak concentration, which the remaining concentration results are calculated relative to.

$Area_{REF}$ = the area of the reference peak in the unknown sample.

$Known\ (St.\ Factor)_{REF}$

= the standard factor of the selected reference peak.

The above peak parameters refer to the unknown sample.

It can be seen from the Job 60 listing in Appendix C that the factors calculated in Job 59 were stored on disk in the Job 60 file.

7.4.4 <u>Off-line Processing Job Results</u>

The results of an off-line processing job are shown in Appendix C. The chromatograph data was obtained in card format and entered into the appropriate file on disk. Since Job 50 was used to analyze the data, the scanning rate could not be altered from 5 points per second in order that the peaks would occur in the correct time bands. However, the user could specify other jobs to handle the off-line processing at different sampling rates.

7.4.5 <u>Repeated Analysis and 'Queuing Several Jobs' Features</u>

The effect of the analyst entries shown in Appendix C.15 to obtain the repeated analysis and 'queuing several jobs' features can be seen in the printout of the results. These results demonstrate the repeated analysis technique with cycling of several jobs, Jobs 50, 51 and 52 being used in this example. A comparison of the results for these jobs has already been given in Section 7.4.3.

7.4.6 Process Control

An example demonstrating the process control loop feature using a process chromatograph as the detecting element is shown by the results of Job 61 in Appendix C. These results show that the measurement of control loop 163 has changed according to the new composition value calculated in the chromatograph analysis.

7.4.7 Repeatability of Chromatograph Results

Job 33, shown in Appendix C, is defined for an actual Beckman GC-2 run using an unknown sample of methyl and ethyl alcohol. The results were obtained by injecting 5 microliters of this unknown sample into the chromatograph. From the Job 33 listing, it is noted that the peaks were given arbitrary standardization factors. Comparing the results of Job 33 with the chromatogram produced, the reader will notice that only two peaks have been reported in the printout while in fact three peaks are shown on the chromatogram. This is explained by the use of a control parameter, shown in the job listing, which caused the scanning routine to idle until the start of the methyl alcohol peak. This job was repeated six times, the results being presented in Table 7.2. These results show quite a variation for the same unknown sample. However, the author feels that some of the variation must have been caused by the analyst techniques such as sample injection etc., since the author makes no claim to be a chromatograph analyst. Table 7.2 also shows the results of six runs using Job 50 with the X-Y recorder and six runs using the off-line processing technique. It is apparent from these results that since no variation was encountered using the same digital input data in the off-line processing technique, the results are repeatable. The variation in the results using the X-Y plotter and the Beckman GC-2 must have been caused by hardware and analyst operating

TABLE 7.2

REPEATABILITY OF COMPUTER CHROMATOGRAPH MONITORING

Beckman GC-2

Beckman GC-2 operating conditions helium flow rate 50 cc/min. helium pressure 18 psig, column temperature 130°C.

	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
% Methyl Alcohol	51.613	52.122	50.862	50.483	48.281	50.114
% Ethyl Alcohol	48.386	47.877	49.137	49.516	51.718	49.885
X-Y Plotter and Job 50						
% Propane	22.491	22.802	22.747	22.494	22.391	22.606
% Butane	27.180	26.939	27.121	27.501	26.578	26.790
% Pentane	50.327	50.257	50.131	50.003	51.029	50.602
Off-line Processing and Job 50						
% Propane	23.130	23.130	23.130	23.130	23.130	23.130
% Butane	27.226	27.226	27.227	27.226	27.226	27.226
% Pentane	49.643	49.643	49.643	49.643	49.643	49.643

techniques.

The computer was checked for accuracy by counting squares under the peaks of the chromatograms to obtain the peak areas for Jobs 20 and 33. The calculated results were found to be approximately the same after normalization.

8. FUTURE WORK

The Gas Chromatograph, Direct Digital Control and Process Operators Console programs were generated using the IBM 1800 Time-Sharing Executive System. This system design necessitated the use of 26,000 words of core for the system skeleton, thereby leaving only 6,767 words for variable core. Since the present disk resident coreloads require the 6,767 words in variable core for execution purposes, the skeleton size cannot be increased. This limitation in skeleton size prevents additional modifications requiring skeleton area from being applied to the system.

IBM have now issued the IBM 1800 Multiprogramming Executive Operating System (MPX), which is designed to asynchronously time-share several independent real-time processes with concurrent background batch processing functions. MPX allows better utilization of the core area, enabling the various coreloads to be assigned to particular core areas.

Multiprogramming is achieved through the employment of the programmed interrupt feature to control the execution of any one of 24 possible core storage areas at any moment in time. The major objective within the MPX design is to keep more of the total system busy by allowing maximum overlap of input/output operations and program execution between the various core areas. For example, the transfer of coreloads from disk to these areas is overlapped with the execution of the highest priority program in core at the particular time. MPX also allows the user to change his system on-line by changing a special coreload which would service interrupts specified for the new system application.

The combined system, GC, DDC, PØC, could be re-designed to operate more efficiently under the MPX system and allow further modifications to be made which could not be handled by TSX. Since MPX distinguishes

priorities on a particular interrupt level, this allows more programs to be specified as interrupt coreloads, which is badly needed with the TSX system. However, since MPX occupies 5000 words more of core area than TSX, some of the GC system skeleton resident routines will have to be specified as coreloads.

One approach would be to place the timer A interrupt servicing programs in the special or SPAR core area, and dedicate one of the partition corelaods for the GC basic cycle routines. The final calculation and report output routines could be specified as interrupt coreloads while the disk maintenance routines could be specified as mainline level. The DDC and PØC programs would have to be modified in a similar manner to operate as queued coreloads in a partition area. Before attempting to implement this MPX system design, careful consideration should be given to the maximum user requirements to be operated simultaneously, so that the sizes of the core areas can be modified accordingly.

9. CONCLUSIONS

The IBM Time Sharing Executive System was used to generate a system which is capable of monitoring and controlling the operation of both laboratory and process gas chromatographs simultaneously with direct digital control of process control loops. The features of the modified GC system have been demonstrated to interested personnel in the Chemical and Petroleum Engineering department at the University of Alberta.

One major feature is the application of the DDC program with the GC system. This combination of programs enables the results of chromatographic analyses to be used as the measured variables for process control loops in the DDC program. To achieve good control, the user must ensure that his chromatograph hardware is capable of providing reproducible results, as compared with the Beckman GC-2 results, shown in Table 7.1.

Another useful feature of the system is the capability to process chromatographic data off-line at a user specified sampling rate, provided the data is stored on disk previously. Presently, the chromatograph data for this feature is collected by a non-process job and punched out in card format, which can then be loaded into the correct disk file. This feature would be useful in a study to determine the sampling rates and smoothing techniques necessary for accurate results.

The IBM 1800 Multiprogramming Executive Operating System is recommended to generate a new combined GC, DDC, PØC system, because of the problems encountered by the author using TSX. One problem using TSX is that the input/output hardware levels occupy the six highest priority interrupt levels. For this reason, any interrupt programs which required input/output operations, such as the typewriters, had to operate on levels lower than level six. This enabled three programmed interrupts to be

specified for the system, since the system has eleven priority levels and the 1816 keyboard interrupt and console interrupt servicing programs occupy one each. MPX allows the user to specify priorities within a priority level, thereby enabling the user to arrange all the input/output hardware on the first two or three highest priority levels, leaving more levels available for programmed interrupts. MPX is also recommended to overcome the problem of no available skeleton space for further modifications.

The author would certainly recommend the use of the modified GC system in the department for laboratory chromatograph monitoring and control studies employing chromatographs in process control loops.

10. NOMENCLATURE TABLE

GC	Gas Chromatograph
PGC	Process Gas Chromatograph
DDC	Direct Digital Control
PØC	Process Operators Console
TSX	Time Sharing Executive System
MPX	Multiprogramming Executive Operating System
ADC	Analog-Digital Converter
DAC	Digital-Analog Converter
DACS	Data Acquisition and Control System
INSKEL COMMON	Permanent Core Common Area
CPU	Central Processing Unit

II. BIBLIOGRAPHY

1. Beckman Industries Corp., Model 320-C Process Gas Chromatograph Instruction Manual, 1965.*

2. Lichtenstein, Ira., "Computer Automation of Chromatographic Data Processing," Electronics Associates Inc., New Jersey.*

3. Mine Safety Appliances Company, Model 550 Process Gas Chromatograph Analyzer Literature.*

4. Purcell, J.E., Condon, R.E., Ettre, L.S., Gas Chromatography Applications, Perkin-Elmer Corp., No. GC-AP-009, March 1967.*

5. Beckman Industries Corp., Model GC-4 Gas Chromatograph Instruction Manual. 1965.*

6. Perkin-Elmer Corp. GC Newsletter, Vol. 2, No. 2, Fall 1965; Vol. 2, No. 3, Fall 1965.*

7. IBM, Gas Chromatograph Monitoring Program, General Program Library, Program No. 23.5.001.*#

8. Dal Nogare, S., Juvet, Jr., R.S., "Gas Liquid Chromatography Theory and Practice," John Wiley, New York (1962).

9. IBM, "Gas Chromatograph Sales Presentation," 1967.*

10. IBM, "Laboratory Chromatograph Monitoring with the IBM 1800," 1967.*

11. Barret, A.T., "The Integrator/Computer System for Gas Chromatography Data Automation," Infotronics Corp. Literature.*

12. Brown, J.E., Chem. Eng. May (1968).

13. Briggs, P.P., Control Eng. September, (1967).

14. Villalobos, R., Chem. Eng. Progr., No. 4, p. 55 (1968).

15. Maley, L.E., Control Eng. November, (1961).

16. Mears, F.C., Hydrocarbon Processing, 46, No. 12, p. 105-8 (1967).

17. Dietz, J.M., API Meeting Philadelphia Preprint No. 33-68, May 1968.

18. Harris, E.B., Hickerson, J.F., Morgen, M.V., "Use of a Real-Time Monitoring Computer for Gas Chromatography and Mass Spectrometry Analysis," Humble Oil Company.*

19. Anon. Chem. Eng. News 45, No. 21, p. 63 (May 15, 1967).

20. Felton, H.R., Hancock, H.A., Knupp Jr., J.L., Instr. and Control Systems, 40(83-5) Aug., 1967.

21. Frazer, J.W., Anal. Chem. 40, No. 8, pp 26A-28A, pp 30A-40A, (July 1968).

22. Anon., Chem. Eng. Progr., 63, No. 4, p. 123 (April 1967).

23. Fraade, D.J., Instr. & Control Systems, 39 (75-6) Aug., 1966.

24. Briggs, P.P. Jr., Laboratory Automation 22nd ISA Annual Conf. Proc., 1967, 22(33-1-AID/CHEMPID-67) (Pt. 4), 10 pp.

25. McCullough, R.D., J. Gas Chromatography, 5 (12), 635-40, 1967.

26. Mears, F.C., Oil and Gas Journal (64), Sept. 26, 1966.

27. Ogle, F.T., Chem. Eng. Progr. No. 61, p. 87-92 (1965).

28. Kuo, B.C., "Analysis and Synthesis of Sampled Data Control Systems," Prentice Hall, Englewood Cliffs, NJ. (1964).

29. Kuo, F.F., Kaiser, J.F., "System Analysis by Digital Computer," John Wiley, New York. (1966).

* Available in the GC File at the University of Alberta Chemical and Petroleum Engineering Department.

Modified by the Author to reflect changes.

APPENDIX A

A.1 Modifications to the system because of hardware changes.

Because the model 1816 typewriter keyboard replaces two model 1092 programmed keyboards the following programs can be omitted from the original IBM system.

PUSH1	LRC1	CLUC1	GDAS1	T921	STRB1	OFF1
PUSH2	LRC2	CLUC2	GDAS2	T922	STRB2	OFF2

The programs which replace the above and service the model 1816 typewriter keyboard are:

KBINT and I1816

These programs store entries made by an analyst through the keyboard into the first 30 words of an array in Inskel Common IN92X(60). For each known chromatograph analysis being made, the analyst would press the keyboard request button and enter the required information as shown in Figure A.1. In this example a '1' is entered first to print out the analyst operating procedures. The minimum cycle time of the supervisory program has been set arbitrarily at 60 seconds. To change this to a lower value the routine I1816 would have to be modified.

The data entered between the brackets for the first set of brackets specifies a code that corresponds to a previously defined job heading. The reader is referenced to Appendix B for greater detail on job heading information. The second set of data specifies that job 20 is to be carried out on chromatograph number 1.

A.2 Reduction in the Number of Monitored Chromatographs.

The procedure to change the number of chromatographs in the GC monitoring program, according to the IBM GC manual (7) is as follows:-

1) change the value of the equate card in the assembler language

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2 CYCLE TIME
ONLY -- 3 OMIT
1
          GC. ANALYST ENTRY PROCEDURES
FIRST ENTRY 1 ROW      15 COLS.
DATA ENTERED BETWEEN THE BRACKETS RT. JUSTIFIED FORMAT(I2)
COLS. 1-- 9 REPRESENT CODED JOB HEADING INFORMATION
RELATED TO DATA ENTERED VIA DISK MAINTENANCE ROUTINES
COL. 10 = LOOP ID. NO FOR THIS GC.
COL.11=ELAPSED TIME BEFORE GC. REINITIATION
=COL. ENTRY * CYCLE TIME OF SUPERVISORY ROUTINE
COL. 12  ENTRY FOR CONTINUOUS SYSTEM-- 1 CONTROL- 2 CONTROL
 WITH PRINTOUT - 3 PRINTOUT
COL. 13,14,15, = SUCCESSIVE JOB NO. TO BE DONE SEQUENTIALLY
2ND. ENTRY 1 ROW , 15 COLS.
COL. 2,3,4, = JOB NO.
COL. 5,6 = GC. NO.
COL. 7,8 = MONTH
COL. 9,10 = DAY
COL. 11,12,13 = TIME
COL.14,15 ANALYST NO.
     PRESENT CYCLE TIME =      USECS.
ENTER NEW CYCLE TIME -- MIN. VALUE 60 SECS. FORMAT(I4)
0060
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( 9)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
      0    5    0    0    1
```

(For further explanation of the second analyst entry, consult the GC Manual (7) pages 214, 215.)

Figure A.1 Model 1816 Typewriter Keyboard Analyst Entry Procedures Listing

symbol table labeled KINUM to the new total of chromatographs. Consult the GC program listing file for a listing of KROM.

2) all variables in Inskel Common dimensioned as 20 should be dimensioned to this new number of chromatographs ie. 6. Also IWORK in Inskel Common should be dimensioned to 70 * XINUM. Similarly, the parameters QO, NMAMP, NINE2, NOENT, NSTAK and MSPEC in the symbol table KROM must be user defined.

The reason why the program would only work for an even number of chromatographs was due to the program SCAN transferring double precision data from an even address location to an odd address location. Only even addresses existed if the number of chromatographs was even. Thus at the time 6 chromatographs was chosen. However to avoid this error the SCAN program data should never be loaded double and stored double, which occur at statements SCAN 791 and 792.

A.3 Allocation of Inskel Common Area.

The following changes were made to leave N (integer constant) words available for other users programs at the beginning of Inskel Common.

1) include an IDUM(N) as the first array in Inskel Common.

2) change the QO equate card (address of Inskel Common +1) to QO = (address of Inskel Common +1) - N. Consult the GC program listing file for program listing KROM.

3) change the following assembler language programs, which use the contents of fixed core address 156 ie. the address of the start of Inskel Common +1, directly. Subtract N immediately following these instructions:

SCAN	Statement numbers 71, 89, 775, 786
KONTL	Statement numbers 46, 99, 111
CONTL	Statement numbers 44

A better way to perform part 3 would be to subtract a variable named for example INDUM after each of these statements and then define INDUM = the value N in the symbol table (KROM). This would then allow these programs to be changed by changing one equate card.

A.4 Combination of the GC, DDC, PØC Systems.

The interrupt coreload situation will be outlined below with reference to both the original and modified systems. With a chromatograph-computer dedicated system the following interrupt coreloads were set up, as specified in the IBM GC Manual (7).

SCAN	programmed interrupt level 2
CI	programmed interrupt level 6
HANDL	programmed interrupt level 7
PANEL	programmed interrupt level 8
DCONT	programmed interrupt level 9
DCODE	programmed interrupt level 10
DIGST	programmed interrupt level 11

process interrupt levels:-

PAN 1	process interrupt level 3/bit 0
PAN 2	process interrupt level 3/bit 1
PAN 3	process interrupt level 3/bit 2

With the combined DDC, PØC, GC system, the DDC - PØC programs required levels 7 and 8. Therefore DCONT and DIGST were made into queued mainline programs, while HANDL, PANEL and DCONT were all placed on one level as one large coreload. Since this interrupt coreload was too large to fit into computer variable core, one small interrupt coreload (HANDL) was made and the other subroutines HDDDL, PANEL and DCONT were brought into the computer when required by using the *LOCAL (load on call) statement.

A programmed interrupt KBINT was also required to service the keyboard interrupt on the model 1816 typewriter keyboard. Another programmed interrupt DWWRT was also specified to enable a disk read to occur when processing the chromatograph raw data from a disk file.

A new list of GC programmed/process interrupt levels is shown below:

DWWRT	programmed interrupt level 3
TNNMM	programmed interrupt level 5
LVL6 (SCAN, SUPVR)	programmed interrupt level 6
HANDL (HDDDL, PANEL, DCONT)	programmed interrupt level 9
CISUB	programmed interrupt level 10
KBINT	programmed interrupt level 11
PAN 1	process interrupt level 3/bit 0

PAN 2 and PAN 3 are no longer required since PAN 1 will handle 16 chromatographs and only 6 are being considered. A more specific outline of the interrupt levels can be seen in the system generation file (13).

The programs TNNMM, LVL6, HDDL are all small modifications outlined in the modified GC Manual (7).

The programs SUPVR and DWWRT are explained in Chapter 4.

The program CISUB replaces CI in order that various programs can be queued from the console interrupt button dependent upon the data switches.

A.5 Supervisory Program.

An overall flow diagram of the routines involved with this GC supervisory program is shown in Figure A.2. The flags that this program checks and sets can be seen in the program listing file under SUPVR in the modified GC Manual (7).

This program could be enlarged upon to perform other GC functions by queuing other programs after some phenomenon has occurred.

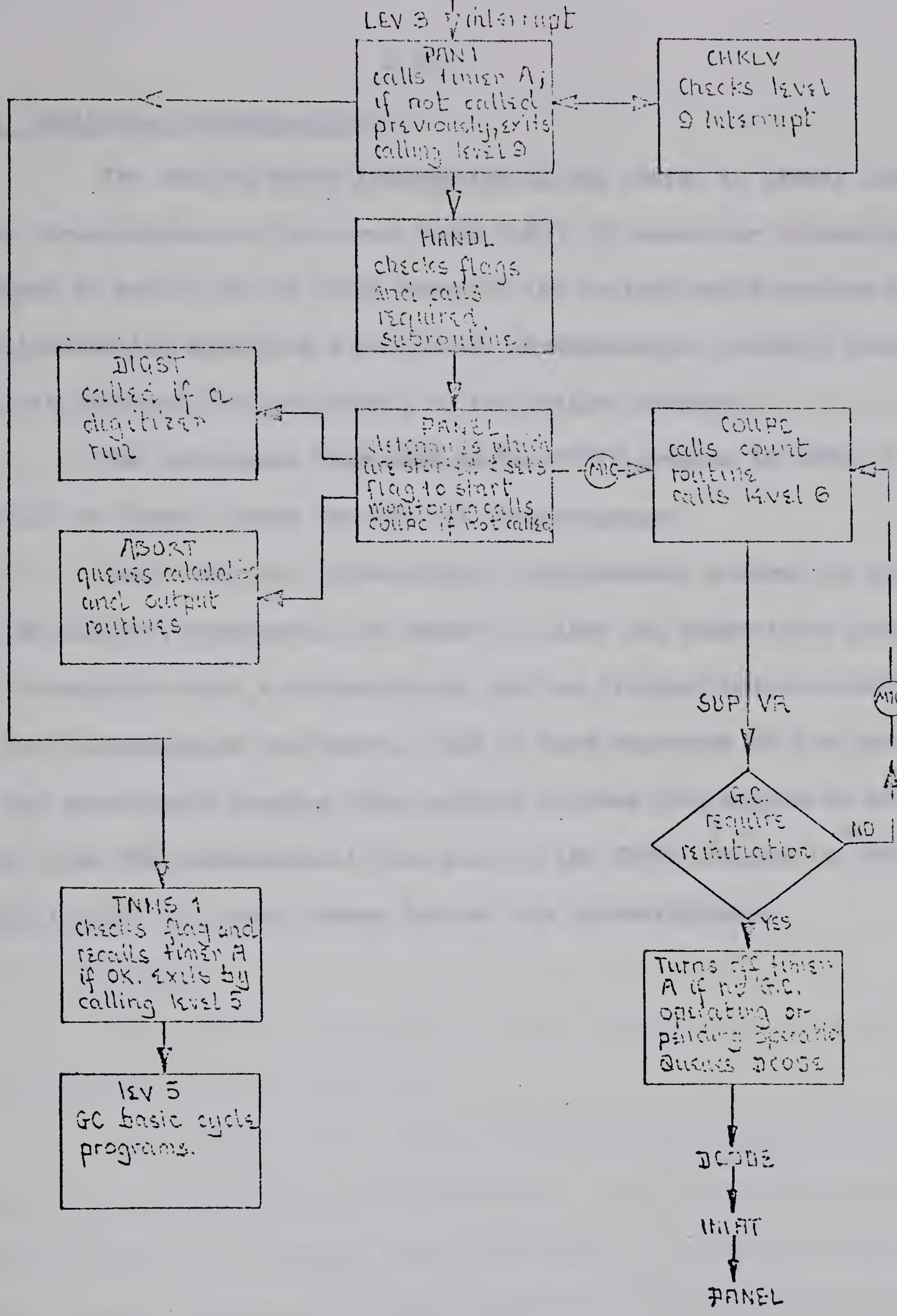


Figure A.2 Overall Flow Diagram of the GC Start Up Routines

A.6 Continuous chromatograph.

The analyst entry information is now stored in Inskel Common for each chromatograph in the array IN18X (180), 30 words per chromatograph. Columns 11 and 12 of the first entry of the analyst entry routine contains the information regarding a continuous chromatograph. Consult section 3.1 of this Appendix for more detail on the analyst entries.

The continuous flag used in the SUPVR program is IN92Q = IN92X (37-42) in Inskel Common for the six chromatographs.

The timing for chromatograph reinitiation, entered in column 11 by the analyst, represents the number of times the supervisory program has to be executed after a chromatograph job has finished before reintiation of the chromatograph can occur. This is then dependant on the cycle time of the supervisory program whose present minimum time equals 60 seconds. This 'time for reinitiation' flag used in the SUPVR program is IN92P ≡ IN92X (31-36) in Inskel Common for the six chromatographs.

APPENDIX B

DISK MAINTENANCE PROCEDURES

This appendix will be presented as a modified version of the Disk Maintenance Procedures outlined in the GC Manual (7). The main changes have been made because the 1092 programmed keyboards have been replaced with an 1816 typewriter keyboard. Because of this change, a first analyst entry via the 1816 keyboard such as:

(8) (9) ( ) ( ) etc.

which utilizes the first two columns, is equivalent to specifying page 8 of the 1092 keymats (see page 196 of the GC Manual (7)) and pressing button number row 9, column 2 etc. Thus when using the DFIN 1092 cards in the following section, by specifying on these cards that page 8, button number row 9, column 2 is a heading such as 'GAS CHROMATOGRAPH MONITORING', the user will obtain this title as a heading to the final report, if the analyst entry is made as shown above.

B.1 Introduction

The procedures described in this section allow the user to perform the following required functions:

1) Define to the computer system a chromatograph technique used with a given chromatograph detector number. This includes such things as concentrations in standards, times that peaks are expected in each chromatogram, response factors to be used, labels to be printed on the analysis reports next to each calculated concentration, and control actions such as ECO operations for column switching and backflushing, and peak detection scan parameter changes.

2) Define variable values such as the reference peak time, or job finish time. The reader is referred to the glossary of terms on page 337 of the GC Manual (7). These values are stored in a table on the disk

and later used as input when a define job function is performed as described above.

3) Define alphanumeric sample description constants associated with the 1816 keyboard entries. (First entry columns 1 - 9)

4) Delete variable values from the tables mentioned above or make the space allocated to a variable available for use.

5) Modify the values of variables used in chromatograph jobs. Modification of a variable used in more than one job can be performed by input of a single card. Up to seven job-chromatograph combinations may use a single variable.

6) List all pertinent information about the value of each variable, its definition status, and job numbers using that variable.

7) List all pertinent information concerning any given job-chromatograph combination.

8) List the alphanumeric sample description constants associated with the 1816 keyboard entries. (First entry columns 1 - 9)

9) Pack all files on the disk to eliminate unused areas, thus minimizing the disk storage requirements.

The define, delete, and modify functions of the disk maintenance programs allow the user to easily define to the system chromatograph procedures without need for reprogramming. The list functions allow the user to retrieve information about chromatograph procedures without the need for vast amounts of external documentation.

The capabilities listed above are executed by loading control cards and data cards filled out by the user into the system. Each primary function to be performed is initiated by input of a control card which may or may not be followed by detail data cards. The primary control card

indicating each major function to be performed will be referred to in this section as the 'control card'. Details on the meanings of the fields in the control card are given in a section below. Figure B.1 is a diagram indicating the sequence in which the control cards and data cards must be loaded. The section below gives a list of the valid primary control card formats and a general description of how each is used.

B.2 Control Card Description.

DFIN VAR:

This card is used to associate a numerical value with a variable number. Each variable number is equal to its record number in File 25 (position in the variable table). Definition of variable values is normally the first function performed before any chromatograph methods are defined or actually executed on line in the monitoring system. All variables used in a job must be defined in the Variable table before the job definition cards are entered into the system. There are six kinds of variables used in chromatograph jobs that are always defined in the variable table before job definition:

1) Chromatogram termination time in seconds.
2) Expected time, in seconds, of the reference peak.
3) Tolerance of reference peak.
4) Low time of each time band.
5) High time of each defined time band.
6) 'time-to-do' of each ECO or change parameter action.

The XFUNC, XSUB, and KARDS fields should be filled in on this card.

DFIN JOB:

This control card indicates to the maintenance program that data cards which follow will define a procedure used by the computer in monitoring

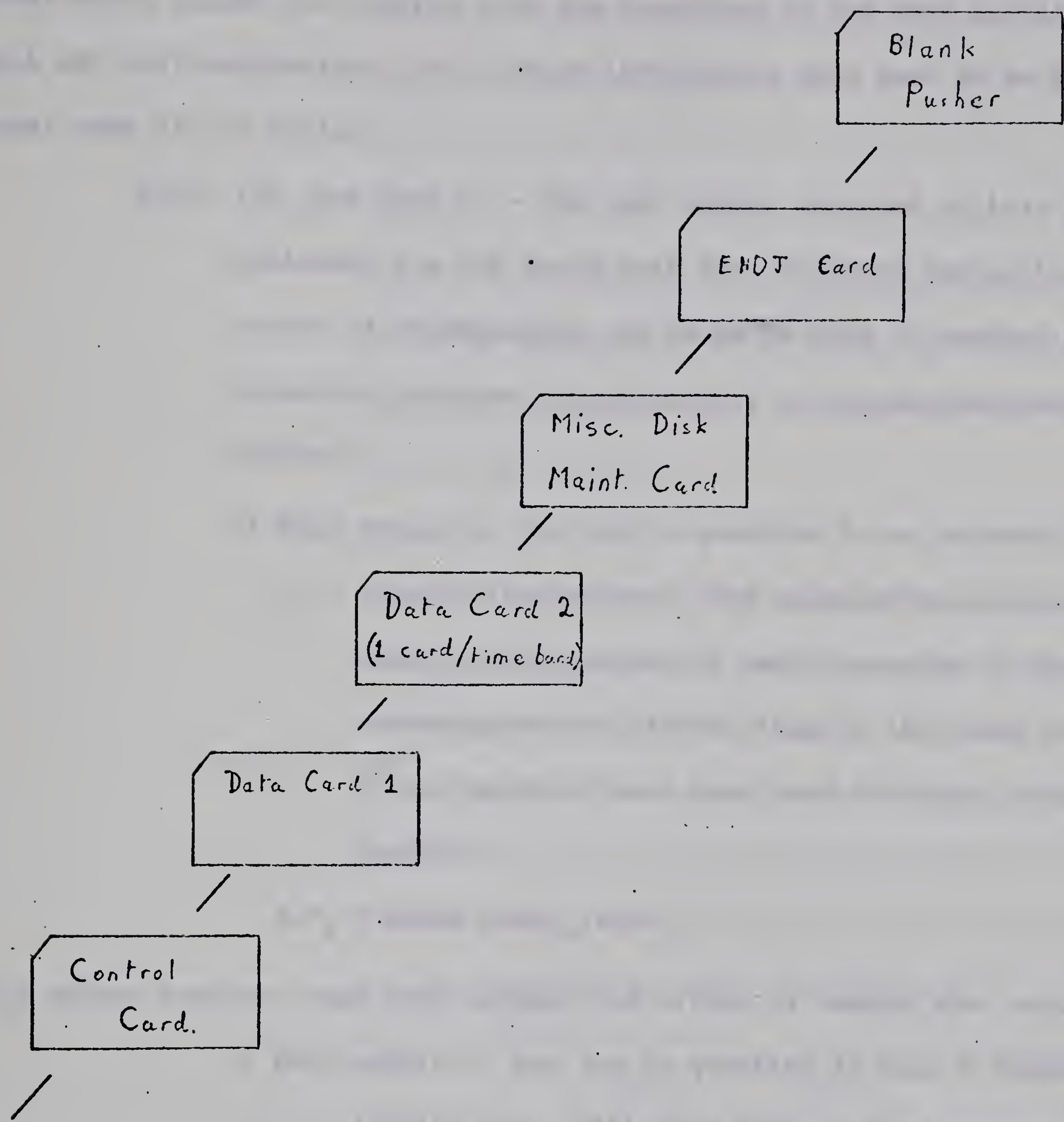


Figure B.1 Disk Maintenance Input Deck

a chromatogram. The fields that must be filled out on the detail data cards which follow the control card are described in the next section. Most are self explanatory, but further information does need to be given about some of the fields:

KALC (On Data Card 1) - The code number inserted in this field indicates how the basic peak data detected during the course of chromatogram run is to be used to produce either calculated unknown concentrations or standardization response factors.

If KALC equals 1, the job in question is an external standardization run. The calculation routine takes known concentrations of peaks expected in the chromatogram and divides them by the total area of all peaks of each time band to obtain response factors.

$$R.F_k = \text{Known Conc.}_k/\text{Area}_k$$

This method does not take into account the effect of sample size variations.

If KALC equals 2, the job in question is also a standardization run. With this method, the response factor of each time band in the chromatogram is divided by the response factor of the reference peak and stored on the disk for later use. This technique tends to eliminate sample size variation problems.

$$R.F._{k(\text{stored})} = R.F_k/R.F._{\text{ref.}}$$

If KALC equals 3, this job calculates unknown concentrations for samples run in the laboratory. The calculation

routine multiplies the areas determined during the course of the chromatogram run by previously stored sensitivity or response factors to obtain the concentrations of the peaks in each time band. The sensitivity factors may have been stored on the disk by a previously run standardization job (see above) or entered into the system in the space provided on Data Card 2 (see section below for Data Card 2 description).

$$\text{Conc.}_k = \text{Area}_k * \text{R.F.}_k$$

If KALC equals 4, this is also an unknown concentration run. The calculation routine for this code is exactly the same as for KALC = 3, except that all calculated concentrations are normalized to a given percentage as entered in the required field on Data Card 1. Unknown peaks (those detected but not expected) are not included in this normalization.

$$\text{Norm. Conc.}_k = \frac{\text{Un-norm Conc.}_k}{\Sigma\,\text{Conc.}_i} \times (\text{Coded Normalization conc.})$$

If KALC equals 5, this is also an unknown concentration run. The calculations made are exactly like the calculations made if KALC equal 4, except that unexpected or unknown peaks are given a response factor of 1.0 and are included in normalization.

If KALC equals 6, this is an internal standardization run. Internal standard response factors are calculated and stored on the disk for later use by a job which

has a KALC equal to 7. The response factors are calculated using the formula below:

$$R.F._k = \frac{\text{Known Conc.}_k * \text{Area}_{REF} * \text{Known R.F.}_{REF}}{\text{Known Conc.}_{REF} * \text{Area}_k}$$

If KALC equals 7, this is an internally standardized unknown concentration run. Concentrations are calculated using the formula given below:

$$\text{Conc.}_k = \frac{\text{Area}_k * R.F._k * \text{Known Conc.}_{REF}}{\text{Area}_{REF} * \text{Known R.F}_{REF}}$$

If KALC equals 9, this is a digitizer run. Only base line corrected areas and absolute retention times (in seconds) of detected peaks are printed out. This is the calculation procedure used when one of two situations exists during a chromatograph run:

1) A 1816 entry was not made before sample was injected into the chromatograph, or

2) For some reason a production run sample needed to be aborted.

IEXP1 and IEXP2 (On Data Card 3) - These two parameters control the amount of exponential smoothing applied to first and second derivatives, respectively, by the SCAN program. As each new DRO value is processed by SCAN, new first and second derivatives are calculated. The formula for calculation of the 'ith' first or second derivative is:

$D(1 \text{ or } 2)_i = (D(1 \text{ or } 2, \text{raw}) - D(1 \text{ or } 2)_{i-1})/X + D(1 \text{ or } 2)_{i-1}$

where:

$D(1 \text{ or } 2)_i$ = newly calculated smoothed first or second derivative.

D(1 or 2, raw) = newly calculated unsmoothed first or second derivative.

$D(1 \text{ or } 2)_{i-1}$ = last smoothed first or second derivative.

X = A smoothing factor determined by the value of IEXP1 or IEXP2. X is equal to 2**IEXP1 or 2**IEXP2.

The values of IEXP1 or IEXP2 can be changed <u>at any time</u> in the course of a chromatogram by including a Data Card 3 for the change parameter action. Normally, the larger the values of IEXP1 and IEXP2 (as peaks flatten out), the first and second derivative deadbands (IHIGH and ILOW on Data Card 3) would be decreased accordingly to allow more sensitivity for slope change detection. The value of IHARD and ISOFT on Data Card 3 should be increased as the peaks become wider and flatten out.

Only the XFUNC and XSUB fields need to be completed on the DFIN JOB Card.

DFIN 1092:

This control card indicates to the disk maintenance program that a series of data cards follow the main control card. Each of the data cards that follow this control card defines an alphanumeric constant associated with a specific 1816 job heading code. The alphanumeric constant defined is the verbage that is printed at the top of the analysis report heading when that code is used prior to injection of a chromatograph sample. The XFUNC, XSUB and KARDS fields must be completed on this card.

DLET VAR:

Entry of this control card causes a variable value to be deleted or placed in an undefined state in File 25. The XFUNC, XSUB, and KVRNO fields must be filled in for this card.

DLET JOB:

Entry of this control card into the system causes all data records on disk to be deleted for a particular job-chromatograph combination. After a delete job function, the previously defined job can no longer be run in the laboratory on the specified chromatograph unless it is redefined and entered into the system. The space made available on certain files by deletion of jobs can later be utilized using the PACK routine described below. The XFUNC, XSUB, MTHNO, and KHRNO fields must be completed on this card.

MOD VAR:

This control card is provided to eliminate the need for deletion and redefinition of an entire job simply because one variable's value needs to be redefined. Entry of this card into the system causes the value of the variable specified to be redefined in all of the jobs which use that variable (7 jobs may use a given variable). XFUNC, XSUB, KVRNO, and VALUE fields must be completed on this card.

LIST VAR:

Entry of this card causes all pertinent information about a variable or group of variables to be listed on the disk maintenance 1053. If only a job number field (MTHNO) is entered in addition to the words LIST VAR, then all variables used by that job number will be listed. If only the chromatograph number field (KHRNO) is filled in (in addition to the words LIST VAR) then only those variables used in association with that

chromatograph number will be listed. If both job number and chromatograph number fields are completed, then only those variables associated with the specified job-chromatograph combination will be listed. If only the variable number field (KVRNO) completed, then only that single specified variable will be listed.

LIST JOB:

Entry of this card causes all pertinent information about a job-chromatograph monitoring technique to be retrieved from the disk and listed on the disk maintenance 1053. XFUNC, XSUB, MTHNO, and KHRNO fields must be completed.

LIST 1092:

Entry of this card indicates to the disk maintenance programs that a series of data cards follows the primary control card. Each data card specifies that the alphanumeric constant associated with a specific 1816 job heading code is to be printed on the disk maintenance 1053. The XFUNC, XSUB, and KARDS fields must be completed.

ENDJ:

Entry of this card into the system indicates to the disk maintenance programs that no more disk maintenance functions follow, and that the job stream is completed. Only XFUNC is filled in.

PACK:

Entry of this card causes the disk maintenance programs to pack all active data files hence making spaces left by deletion of jobs available for use. Only XFUNC is filled in.

B.3 Card Handling Procedures.

Given below is a step by step description of how the above described control cards and data cards are entered into the 1800 system.

1) Remove all cards from the 1442 card read hopper.

2) Non-Process Run Out any cards inside the 1442.

3) Load required disk maintenance cards in card hopper making sure that last two cards are an ENDJ card and a blank pusher card.

4) Press reader start on 1442.

5) Insure that sense switch 7 on 1800 console is off, (down).

6) Press console interrupt button with data switch 8 on.

7) Disk maintenance programs will then be queued and executed. The contents of each primary control card will be printed out and the 1053 output will indicate to the user the progress as the job stream is processed.

<u>B.4 Disk Maintenance Cards.</u>

<u>Control Card</u>

XFUNC - A four character field designating the desired function.

- DFIN - Define a variable; job or 1092
- DLET - Delete a variable, or job
- MODb* - Modify a variable
- LIST - List pertinent information about a job, variable, or 1092 button
- ENDJ - Ends all disk maintenance until Console Interrupt button is pushed again
- PACK - Repacks all files

XSUB - A four character field indicating whether the function is to be done for a variable, job or 1092.

- VARb - The function will be concerned with listing, defining, deleting or modifying a variable.
- JOBb - The function will be concerned with a job definition,

listing or deletion.

1092 - Either an alphanumeric constant will be defined and associated with a 1816 entry or an alphanumeric constant associated with a 1816 entry will be listed.

MTHNO - Job number right justified in field.

KHRNO - Chromatograph number right justified in field.

KVRNO - Number of the variable to be modified right justified in field.

VALUE - New value of the variable. The format is $x_\wedge xxxxx \pm YY$ where the implied decimal point is to the right of the high order digit and YY is the exponent of the number base 10.

KARDS - The number of cards following.

<u>Data Card 1</u> (One prepared for each job defined)

JOBNO - A three digit number designating the particular chromatograph handling procedure. The things that can be defined for any procedure are listed below. The words 'job number' and 'method number' are synonymous in this documentation. Right justify.

IEXPK - A three digit number designating the number of time bands in a job. This is <u>always</u> equal to the number of Data cards (type 2) in the job definition deck. Right justify.

IXTRA - A three digit number designating the number of extra (unknown) peaks to be allowed in a job. The number of spaces reserved on the disk (File 1) when the job is run on the computer is equal to IEXPK & IXTRA. Right justify.

KALC - A two digit number specifying the calculation procedure to be used. Right justify.

*Note: - A lower case 'b' in a symbol name means that column is to be left blank.

XNORM - The normalization constant for this job. The format is $X_{\wedge}XXX \pm YY$ where the decimal point is one position to the right of the high order digit and YY is the exponent of the number, base 10. All concentrations will be normalized to XNORM when the chromatogram is run under computer control

CNREF - Concentration of the reference peak in any units. The format is $X_{\wedge}XXX \pm YY$ where the decimal is implied one place to the right of the high order digit and YY is the exponent base 10. Used only for internal standards and unknown concentration calculations using response factors calculated using internal standards.

FACRF - Response factor of the reference peak. The format is $X_{\wedge}XXX \pm YY$ where the implied decimal point is one place to the right of the high order digit and YY is the exponent base 10. Used only when CNREF is used.

LASSC - Job number using the factors stored on the disk by this standard. Right justify.

KONTO - Punch 9 if the job requires

1) A backflush operation
2) Column switching
3) Ignore part of scan
4) Change scan variables from the standard set of parameters used in the beginning of all chromatograms. Otherwise enter a 0.

KOORD - Final job number using data for calculation. Used only when more than one chromatogram has to be run to produce a final sample analysis report. Any calculation routine needed for a situation

like this must be provided by the user. The results of the individual chromatograms will always be stored on the disk in Files 1 and 3. The users calculation routine will be called by TAIL when KOORD is non-zero. If this procedure is not required, enter a 0. Right justify.

KRONO - GC number. Right justify.

TMREF - Variable number (in File 25) of the time in seconds of the reference peak. Right justify.

TOLRF - Variable number (in File 25) of the tolerance on TMREF in fraction of TMREF. Right justify. Expected time band of reference peak will be TMREF $\pm$ TMREF * TOLRF. Right justify.

FINTM - Variable number (in File 25) of the finish time of the job in seconds. Right justify. This will be converted to relative time by the disk maintenance program.

IOVER - Logical unit number report should be typed on. Right justify. This logical unit number will over-ride the table look-up (in File 11) normally done in CALC to find the logical unit physically nearest the chromatograph that will be used for the analysis report. Enter a 0 if normal table look-up is desired.

Data Card 2 (One prepared for each time band)

COMPN - Alphameric name to be used for component or group of components which will be printed on the report. Left justify.

SEQ.NO - The time band sequence number. Right justify. The concetrations of all time bands with the same sequence number will be added together and reported as one result. More than one peak can be in a single time band. All peaks in a given time band must have the same response factor. Sequence numbers should

start at 1 and continue upward incremented by one unless a repeat is required for grouping purposes.

JTYPE - Integer representing the type of area calculation to be performed. Right justify. (See figure B.2)

IPLUS - Time band sequence number where triangular area from slice is added from the option JTYPE = 4 is used. Right justify.

FACTR - Response factor used for this time band. The format is $X_{\wedge}XXX \pm YY$ with the implied decimal point to the right of the high order digit and YY is the exponent base 10. Only filled in when the job is an unknown sample calculation as opposed to a standardization run and when literature response factors are used.

COND - Known Concentration in the standard sample. The format is $X_{\wedge}XXX \pm YY$ where the implied decimal point is one digit to the right of the high order digit and YY is the exponent base 10. Only filled in for standardization jobs.

INREF - Punch 9 if this is the reference peak. Otherwise, leave blank.

BOBS - Alphameric units of components in time band to be printed on report. Left justify.

KLOTM - Variable number (in File 25) for the minimum expected time in seconds for the peak or group of peaks in the time band. This time in seconds is converted to relative retention time by the disk maintenance program. Right justify.

HITIM - Variable number (in File 25) for the maximum expected time in seconds for the peak or group of peaks in the time band. This time is also converted to relative retention time. Right justify.

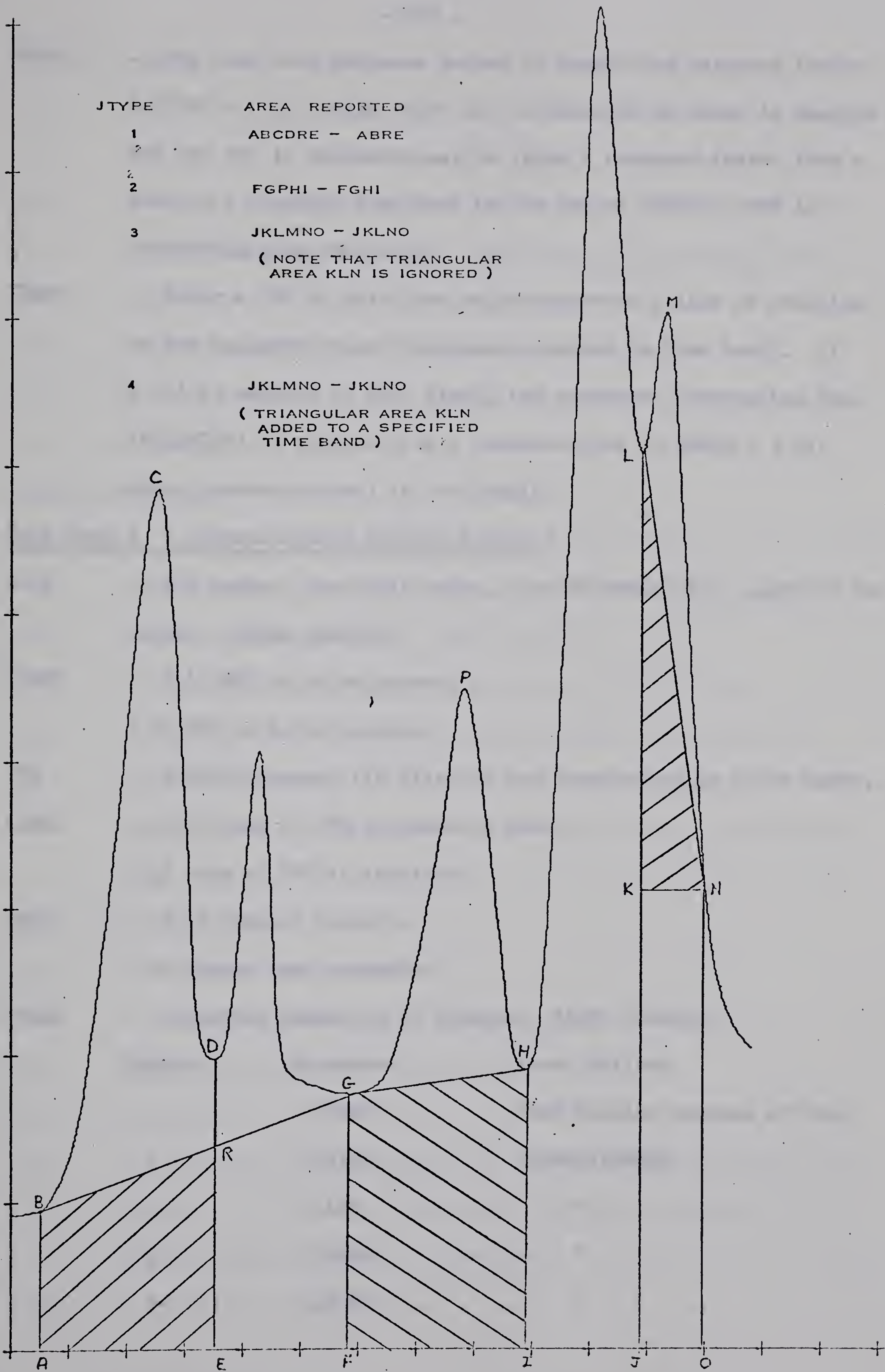


Figure B.2 Peak Area Calculation Options Diagram

IFCGO - The time band sequence number to obtain the response factor if CONC = 0.0. Peaks which may be expected to occur in samples but are not in standards may be given a response factor from a peak in a standard time band by the use of IFCGO. Used in standardization runs only.

IREST - Enter a '9' if this card only represents a line of printing on the analysis report (no peaks expected in time band). If a '9' is entered in this field, the component description (eg. 'BALANCE') is printed with a concentration (= XNORM - Σ all other concentrations) in the sample.

<u>Data Card 3 - Chromatograph Control Record</u> *

ECOX - ECO number, two digit entry. See GC Manual (7), page 219 for values. Right justify.

IONOF - 0 if ECO is to be opened.
1 if ECO is to be closed.

IVR - Variable number (in File 25) for time action is to be taken.

LABRL - 0 if time of IVR is relative time.
1 if time of IVR is absolute.

IWHC - 0 if contact operate.
1 if change scan parameter.

IPARA - Parameter number to be changed. Right justify.

Number	Parameter	Where Defined
1	ISTAT	SCAN Routine program writeup
2	IHIGH	COMMON/INSKEL
3	ILOW	"
4	IHARD	"
5	ISOFT	"

Number	Parameter	Where Defined
12	IEXPI	COMMON/INSKEL
13	IEXP2	"

LWVAL - New scan parameter value. The format is XX^XXX ± YY where the implied decimal point is two digits to the right of the high order digit and YY is the exponent base 10.

ILAST - Punch 9 if this is the last control action.

NOTE * Only used if KONTO in Data Card #1 is non-zero. One card per action frame required.

Variable Data Card

IVRNO - Variable code number equal to the record number of the variable table (File 25) for the entry being defined. Right justify.

KROM - Chromatograph number. Right justify.

ALPHA - Alphameric description of variable. Left justify.

IMAN - The value of the variable being defined. The format is X XXX ± YY where the implied decimal point is one position to the right of the high order digit and YY is the exponent base 10.

Define 1092 Entry Card

NO92 - Punch 1. Right justify.

IPAGE - Page number of entry. Right justify.

ICOL - Column number of entry. Right justify.

NOBUT - Row number of the button defined (0-9).

ALPHA - Alphameric label. Thirty-two characters maximum left justified. See section on 1092 data format for individual 1092 fields' maximum number of characters.

ICON - Punch 9 if last card.

List 1092 Data Card

NO92 - Punch 1. Right justify.

IPAGE - Page number to be listed. Right justify.

ICOL - Column number of entry.

NOBUT - Row number of the button to be listed. (0-9) Right justify.

ICON - Punch 9 if this is the last card.

Digitizer Run. Purpose and Procedure

The object of a digitizer run is to provide peak data on samples about which nothing is known. The sample is injected into the chromatograph and the chromatograph start button is pushed, but no entry is made on the 1816. Under these circumstances, the computer assumes a digitizer run is to be made. It records and prints elution time and area of each peak as a fraction of the total area of the chromatogram. The run is terminated by the chromatograph operator pressing the chromatograph start button again. This causes the computer to stop taking data and begin printout.

However, the computer makes no distinction between one type of job and another except as defined in the job specification card. Thus, a job is defined with special characteristics to give a digitizer run printout.

The input card sequence is as follows. The disk maintenance control card defining a required variable is first. Then four variable data cards follow.

With the variables defined, a new disk maintenance control card follows defining the job. Finally, disk maintenance data cards 1 and 2 define the parameters for the job. Explanation of the card layouts follows.

Disk Maintenance Control Card

Columns	Data	
1-4	DFIN	Prepares the computer to accept a definition.
8-13	VAR	Variables are to be defined.
51	5	Five more variable data cards will follow this one.

Disk Maintenance Data Card 1

Columns	Data	
1-3	008	The job number for this digitizer run.
6	1	There will be only one time band.
7-9	99	Allowance is made for 99 peaks. It could be larger or smaller.
11	9	This sets KALC = 9 and calls for STAN5 to do the peak analysis. STAN5 was designed for digitizer runs. There is no normalization constant, concentration of a reference peak, or response factor for reference peak since none of these are known. Likewise, there is no job number using this standard.
36	0	This job requires no backflushing, column switching or ignoring any part of scan. There is no final job using this data.
40-41	17	The chromatograph number is 17. Actually this field can contain <u>any</u> valid chromatograph number.
46	1	The time to the reference peak is given the variable number 1 which is essentially 10^{26} seconds. In short, this 'reference peak' will never happen.

Columns	Data	
51	2	The tolerance in the reference time is essentially 0.
56	3	The expected finish time for this run is essentially 10^{28} seconds and again we don't ever expect that to happen. (It will in about 10^{21} years.)
57-58		Blank because the output report should <u>always</u> be printed on the 1053 <u>nearest</u> the chromatograph.

<u>Disk Maintenance Data Card 2</u>

Columns	Number	
1-15	Component X	This name is used for a dummy reference peak. It will not be on the report because we will never get to it.
18	1	The digitizer run has only one time band, namely, 1.
20	1	Each peak is to have its area calculated by having perpendiculars dropped from the bottom of the peak to the baseline. (See diagram JTYPE = 1 in Disk Maintenance Procedure Card Layout discussion, Figure C.1.)
23	1	Actually a dummy entry.
24-30	1000+00	A response factor of 1. 00 is given to all peaks.
38	9	For reference peak.
53	4	The earliest we expect the reference peak is a few billion years ahead of the normally expected time.

Columns	Number	
58	5	The latest time the 'reference peak' is expected is the maximum amount of the variable.
61	1	A dummy entry.
62	0	Since all of these peaks are unknowns they will add to 100%, so there is no need to normalize results, and no place to do so because there is only one time band.

Variable Data Card

Columns	Data	
5		This column contains the number of the variable to be modified.
6-7	17	Chromatograph number.
8-22		Dummy description of each variable.
23-29		Actual value of the variable. Reasons for these choices are explained above.

APPENDIX C

RESULTS DEMONSTRATING THE FEATURES OF THE GC MONITORING PROGRAM

This appendix contains the documentation and computer outputs from all the examples referred to and discussed in Chapter 7. The actual computer outputs are presented in the results to show the format of the chromatograph reports. Results from each chromatograph job have been presented with a computer printout of the analysis method used. The information provided on this printout corresponds with the data entered on the job definition cards for the particular method.

The author used two computer input data techniques to obtain these results:

1) the computer input data was provided by an X-Y recorder with curve follower technique using the chromatograms shown in Figure C.1 and C.2.

2) the computer input data was provided by a Beckman GC-2 laboratory chromatograph.

A complete listing of the analyst operating procedures, shown in section C.1, is repeated in this Appendix for completeness. An index of the various job technique combinations is shown in Table C.1.

Table C.1 Index of the Various Job Results

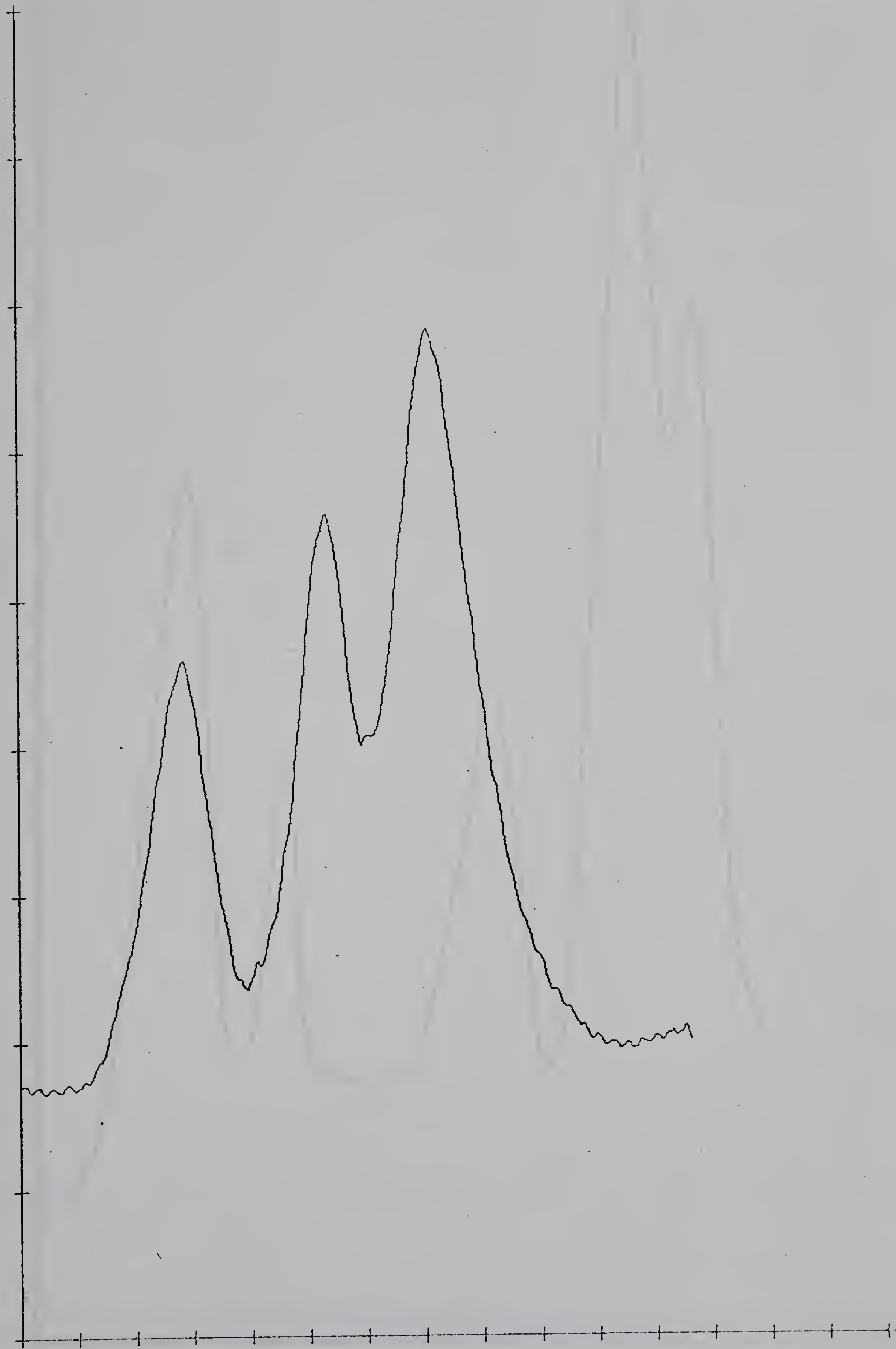
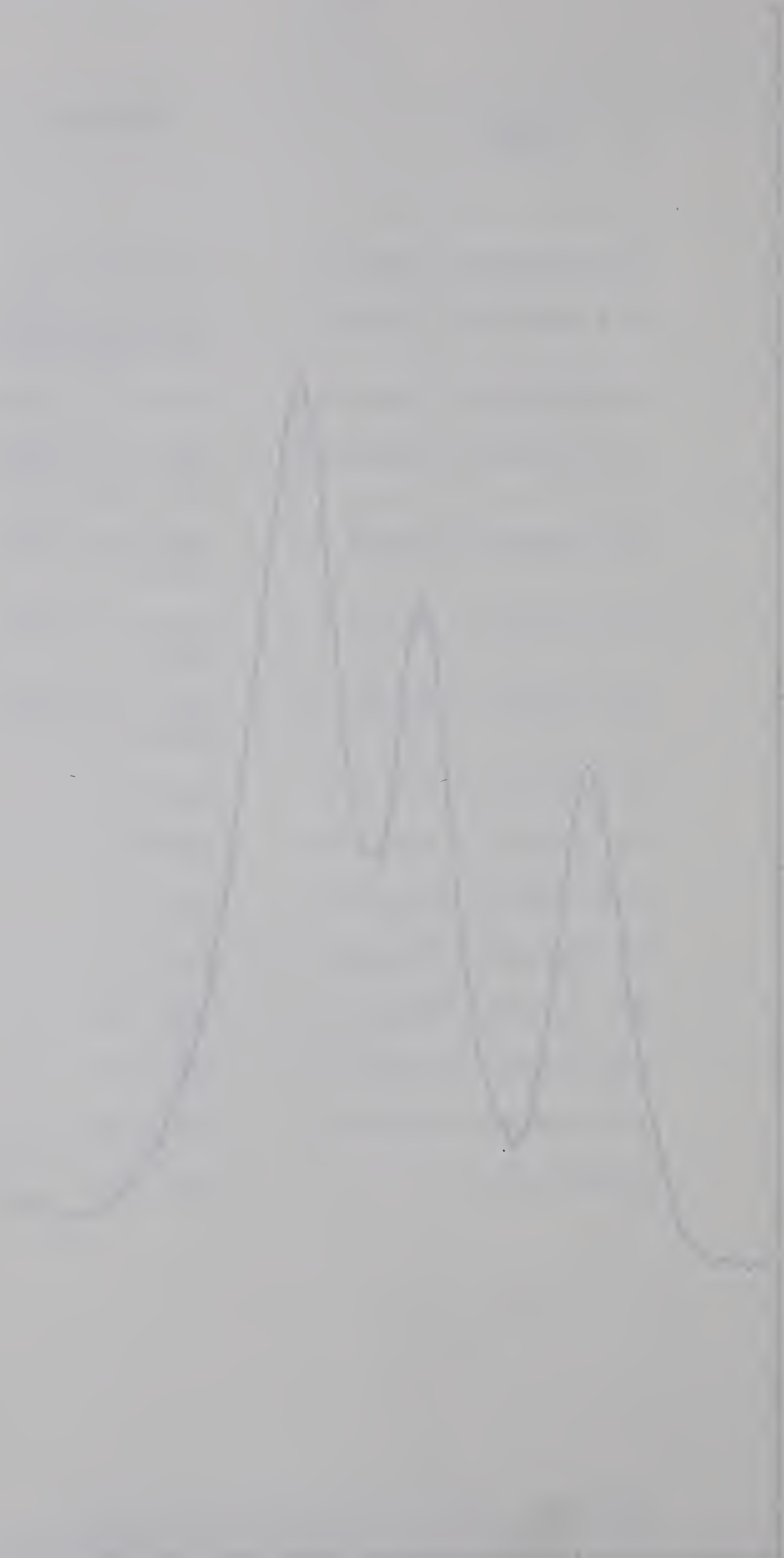


Figure C.1 X-Y Recorder Chromatogram Number 1



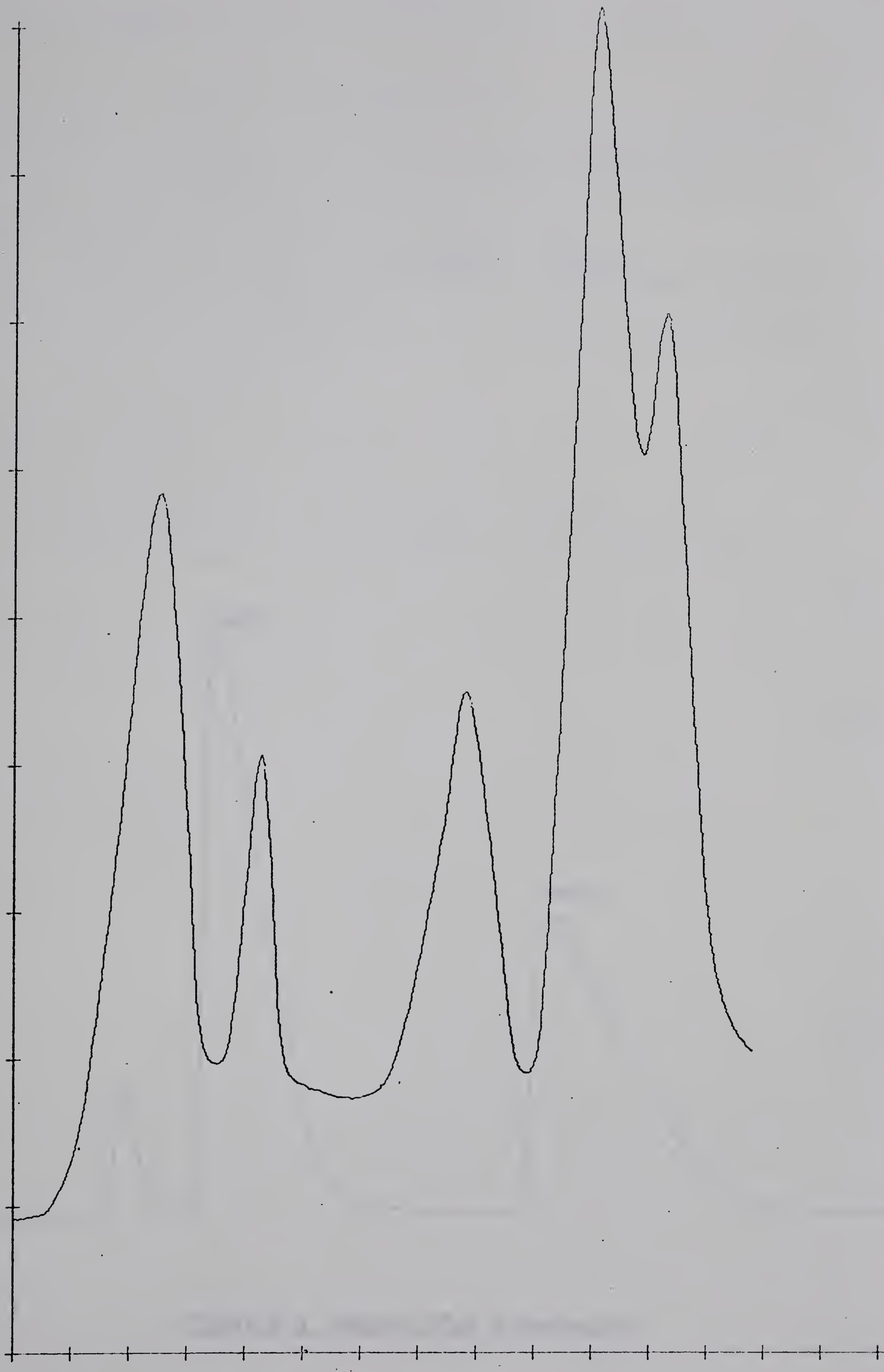


Figure C.2 Recorder Chromatogram Number 2

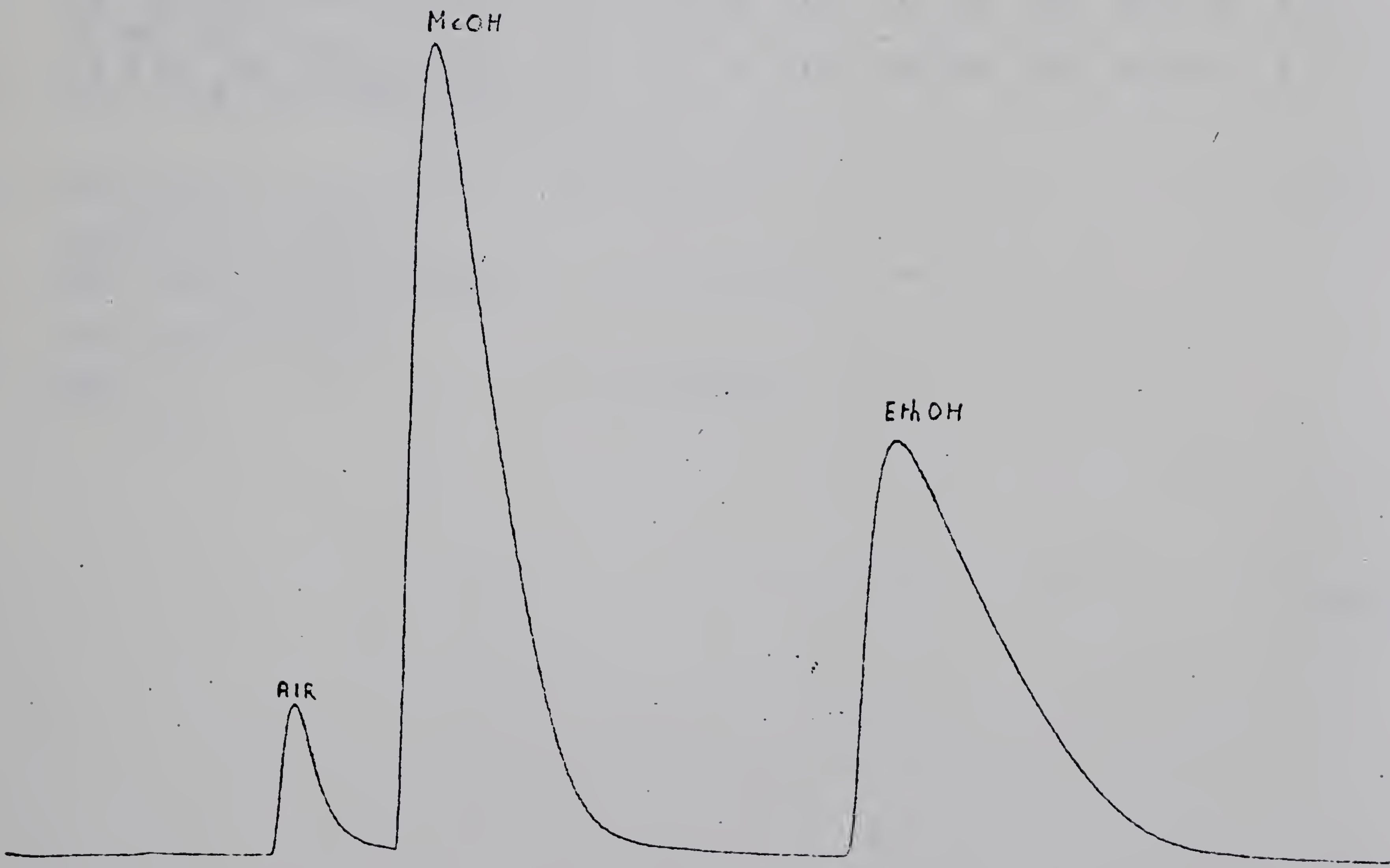


Figure C.3 Beckman GC-2 Chromatogram

C.1

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2 CYCLE TIME
ONLY -- 3 OMIT
1
          GC. ANALYST ENTRY PROCEDURES
FIRST ENTRY 1 ROW       15 COLS.
DATA ENTERED BETWEEN THE BRACKETS RT. JUSTIFIED FORMAT(I2)
COLS. 1-- 9 REPRESENT CODED JOB HEADING INFORMATION
RELATED TO DATA ENTERED VIA DISK MAINTENANCE ROUTINES
COL. 10 = LOOP ID. NO FOR THIS GC.
COL.11=ELAPSED TIME BEFORE GC. REINITIATION
=COL. ENTRY * CYCLE TIME OF SUPERVISORY ROUTINE
COL. 12  ENTRY FOR CONTINUOUS SYSTEM-- 1 CONTROL- 2 CONTROL
  WITH PRINTOUT - 3 PRINTOUT
COL. 13,14,15, = SUCCESSIVE JOB NO. TO BE DONE SEQUENTIALLY
2ND. ENTRY 1 ROW , 15 COLS.
COL. 2,3,4, = JOB NO.
COL. 5,6 = GC. NO.
COL. 7,8 = MONTH
COL. 9,10 = DAY
COL. 11,12,13 = TIME
COL.14,15 ANALYST NO.
     PRESENT CYCLE TIME =     30SECS.
ENTER NEW CYCLE TIME -- MIN. VALUE 60 SECS. FORMAT(I4)
0030
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
 8  9
( J)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
    0   5   0   0   1
```

C.2

```
LIST   JOB        8       1       0  0.00000000E 00       0
JOB NO. =    8
CHROM. NO. =    1
ANAL. CALC. NO. =  9
NO. OF TIME BANDS + SPACE FOR UNK. PKS. = 100
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.00000000E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.99999159E 26
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=      1
CNTRL DATA 1ST FILE REC. NO. =    1


                      TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND    FOR STANDARD                CALC. CODE        NAME
0.100000E 00 0.999999E 01 0.000000E 00 0.100000E 01                1  COMPONENT X

  COMPONENT    SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME        NO. TO SEQ. NO.  SEQ. NO.  RF. PK.  CALC.CONC.    USED
                                                     BY DIFF.
OMPONENT X        1      1            1        9         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =      1

REF. PK. TIME TOL. VAR. NO. =     2

REL. TIME JOB FIN. VAR. NO. =     3
                      TIME BAND VARIABLE NO. DATA

TIME BAND NO.   REL. LOW TIME   REL. HIGH TIME
     1                4               5
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH = 99.9990
.
THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

DIGITIZER RUN - INST. NO / 1

ELUTION TIME	AREA
19	0.23186997E 00
27	0.27118935E 00
33	0.49694085E 00

Digitizer Run Job Definition Cards

```
DFIN   VAR                                                            5
0000101REF. TIME       1000+26
0000201TOLERANCE       0000+00
0000301FINISH TIM      1000+28
0000401MIN TIM BND     1000+25
0000501MAX TIM BND     1000+27
```

```
DFIN   JOB
008  1 99 9                            0   01     1     2     304
COMPONENT X       1 1  11000+00          9                  4     5   10
ENDJ
```

C.3

```
LIST   JOB        20       1       0  0.00000000E 00       0
JOB NO. =  20
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =   5
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.50000023E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.29999992E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=      6
CNTRL DATA 1ST FILE REC. NO. =  14


                       TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND    FOR STANDARD                 CALC. CODE       NAME
0.500000E 00 0.116666E 01 0.000000E 00 0.998999E 01               1   BENZENE
0.116666E 01 0.156666E 01 0.000000E 00 0.998999E 01               1   TOLUENE
0.156666E 01 0.213333E 01 0.000000E 00 0.998999E 01               1   ALCOHOL
0.213333E 01 0.250000E 01 0.000000E 00 0.998999E 01               1   WATER
0.250000E 01 0.300000E 01 0.000000E 00 0.998999E 01               1   GLYCOL

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME        NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.    USED
                                                      BY DIFF.
BENZENE          1       0          0          9         0
TOLUENE          2       0          0          0         0
ALCOHOL          3       0          0          0         0
WATER            4       0          0          0         0
GLYCOL           5       0          0          0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     50

REF. PK. TIME TOL. VAR. NO. =     51

REL. TIME JOB FIN. VAR. NO. =     52

                       TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME   REL. HIGH TIME
      1               53               54
      2               55               56
      3               57               58
      4               59               60
      5               61               62
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  3.1660

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
 8   9
( ) )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0    2    0    0    1    2    0    0    4    1    4    0    0    1
```

```
* GAS CHROMATOGRAPH MONITORING *          ANALYST NO.   1
1400   8/ 4/68                            JOB NO.      20
                                          INST. NO.     1
```

PERCENT OF TOTAL AREA UNKNOWN = 0.00

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
BENZENE	23.947	%	31	0.2184428E 06
TOLUENE	8.683	%	41	0.7920362E 05
ALCOHOL	14.716	%	60	0.1342413E 06
WATER	35.321	%	73	0.3221923E 06
GLYCOL	17.330	%	79	0.1580865E 06

JOB COMPLETE

C.4

```
LIST   JOB        50       1       0  0.00000000E 00       0
JOB NO. =  50
CHROM. NO. =    1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    3
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999996E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=     11
CNTRL DATA 1ST FILE REC. NO. =  17
```

TIME BAND DATA

LOW LIMIT TIME BAND	HI LIMIT TIME BAND	CONCENTRATION FOR STANDARD	FACTOR	AREA CALC. CODE	COMPONENT NAME
0.700000E 00	0.119999E 01	0.000000E 00	0.100000E 01	1	PROPANE
0.115000E 01	0.155000E 01	0.000000E 00	0.100000E 01	1	BUTANE
0.150000E 01	0.225000E 01	0.000000E 00	0.100000E 01	1	PENTANE

COMPONENT NAME	SEQ. NO.	ADD AREA TO SEQ. NO.	FACTOR FROM SEQ. NO.	NINE = RF. PK.	POS. NO.- CALC.CONC. BY DIFF.	UNITS USED
PROPANE	1	0	0	9	0	
BUTANE	2	0	0	0	0	
PENTANE	3	0	0	0	0	

VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =   10

REF. PK. TIME TOL. VAR. NO. =   11

REL. TIME JOB FIN. VAR. NO. =   12

TIME BAND VARIABLE NO. DATA

TIME BAND NO.	REL. LOW TIME	REL. HIGH TIME
1	13	14
2	15	16
3	17	18

```
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ              0        0       0  0.00000000E 00       0
END OF JOB
```

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( ) )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   0   0   1
```

```
* GAS CHROMATOGRAPH MONITORING *        ANALYST NO.    0
0000  20/ 0/68                          JOB NO.     50
                                        INST. NO.    1

PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	22.491	%	19	0.5814870E 05
BUTANE	27.180	%	27	0.7027151E 05
PENTANE	50.327	%	33	0.1301142E 06

JOB COMPLETE

Unknown Run Job Definition Cards

```
DFIN   VAR                                                              13
   5001REF. TIME        3000+01
   5101TOLERANCE        5000-01
   5201FIN. TIME        9500+01
   5301MIN. TME 1       1500+01
   5401MAX TME 1        3500+01
   5501MIN TME 2        3500+01
   5601MAX TME 2        4700+01
   5701MIN TME 3        4700+01
   5801MAX TME 3        6400+01
   5901MIN TME 4        6400+01
   6001MAX TME 4        7500+01
   6101MIN TME 5        7500+01
   6201MAX TME 5        9000+01


DFIN   JOB
050   3  0 41000+02                           01    10    11    12 4
 PROPANE            1 1   1000+00          9 (               13    14
 BUTANE             2 1   1000+00            (               15    16
 PENTANE            3 1   1000+00            (               17    18
ENDJ
```

C.5

```
LIST   JOB       51      1      0  0.00000000E 00       0
JOB NO. =  51
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =   3
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.27999992E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=     18
CNTRL DATA 1ST FILE REC. NO. =  30

                      TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION    FACTOR            AREA         COMPONENT
 TIME BAND     TIME BAND     FOR STANDARD                   CALC. CODE         NAME
0.500000E 00 0.857143E 00 0.000000E 00 0.100000E 01                  1     PROPANE
0.821428E 00 0.110714E 01 0.000000E 00 0.100000E 01                  1     BUTANE
0.107142E 01 0.160714E 01 0.000000E 00 0.100000E 01                  1     PENTANE

  COMPONENT     SEQ.  ADD AREA   FACTOR FROM   NINE =    POS. NO.-     UNITS
    NAME        NO. TO SEQ. NO.   SEQ. NO.   RF. PK.  CALC.CONC.    USED
                                                        BY DIFF.
PROPANE           1      0            0          0          0
BUTANE            2      0            0          9          0
PENTANE           3      0            0          0          0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     19

REF. PK. TIME TOL. VAR. NO. =     11

REL. TIME JOB FIN. VAR. NO. =     12

                      TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1                13                14
      2                15                16
      3                17                18
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  1.7851

THIS FUNCTION COMPLETE

ENDJ              0      0      0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( J )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   1   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *        ANALYST NO.    0
 0000  20/ 0/68                          JOB NO.     51
                                         INST. NO.    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	22.986	%	21	0.5807246E 05
BUTANE	27.128	%	23	0.6855633E 05
PENTANE	49.884	%	35	0.1260273E 06

JOB COMPLETE

C.6

LIST JOB 52 1 0 0.00000000E 00 0
JOB NO. = 52
CHROM. NO. = 1
ANAL. CALC. NO. = 4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. = 3
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999999E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) = 0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) = 4
TIME BAND DATA 1ST FILE REC. NO.= 21
CNTRL DATA 1ST FILE REC. NO. = 33

TIME BAND DATA

LOW LIMIT TIME BAND	HI LIMIT TIME BAND	CONCENTRATION FOR STANDARD	FACTOR	AREA CALC. CODE	COMPONENT NAME
0.700000E 00	0.119999E 01	0.000000E 00	0.100000E 01	1	PROPANE
0.115000E 01	0.155000E 01	0.000000E 00	0.100000E 01	2	BUTANE
0.150000E 01	0.225000E 01	0.000000E 00	0.100000E 01	1	PENTANE

COMPONENT NAME	SEQ. NO.	ADD AREA TO SEQ. NO.	FACTOR FROM SEQ. NO.	NINE = RF. PK.	POS. NO.- CALC.CONC. BY DIFF.	UNITS USED
PROPANE	1	0	0	0	0	
BUTANE	2	0	0	9	0	
PENTANE	3	0	0	0	0	

VAR. NO. INFO.-

REF. PK. EXPECT. TIME VAR. NO. = 10

REF. PK. TIME TOL. VAR. NO. = 11

REL. TIME JOB FIN. VAR. NO. = 12

TIME BAND VARIABLE NO. DATA

TIME BAND NO.	REL. LOW TIME	REL. HIGH TIME
1	13	14
2	15	16
3	17	18

NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH = 2.5000

THIS FUNCTION COMPLETE

ENDJ 0 0 0 0.00000000E 00 0
END OF JOB

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
 8   9
( ) )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
    0   5   2   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *       ANALYST NO.   0
 0000  20/ 0/68                         JOB NO.      52
                                        INST. NO.     1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	27.165	%	19	0.5611554E 05
BUTANE	13.656	%	27	0.2821100E 05
PENTANE	59.178	%	35	0.1222462E 06

JOB COMPLETE

C.7

```
LIST   JOB        23       1       0  0.00000000E 00       0
JOB NO. =  23
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    5
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.50000023E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.29999992E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=     24
CNTRL DATA 1ST FILE REC. NO. =  36

                      TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND    FOR STANDARD                 CALC. CODE       NAME
0.500000E 00 0.116666E 01 0.000000E 00 0.998999E 01              1   BENZENE
0.116666E 01 0.156666E 01 0.000000E 00 0.998999E 01              1   TOLUENE
0.156666E 01 0.213333E 01 0.000000E 00 0.998999E 01              1   ALCOHOL
0.213333E 01 0.250000E 01 0.000000E 00 0.998999E 01              1   WATER
0.250000E 01 0.300000E 01 0.000000E 00 0.998999E 01              3   GLYCOL

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.   USED
                                                        BY DIFF.
BENZENE           1      0           0          9         0
TOLUENE           2      0           0          0         0
ALCOHOL           3      0           0          0         0
WATER             4      0           0          0         0
GLYCOL            5      0           0          0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     50

REF. PK. TIME TOL. VAR. NO. =     51

REL. TIME JOB FIN. VAR. NO. =     52

                      TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1               53               54
      2               55               56
      3               57               58
      4               59               60
      5               61               62
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  3.1660

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
  8    9
(  4)(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
       0    2    3    0    1
```

* GAS CHROMATOGRAPH MONITORING * ANALYST NO. 0
0000 20/ 0/68 JOB NO / 25
INST. NO. 1

PERCENT OF TOTAL AREA UNKNOWN = 0.00

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
BENZENE	28.780	%	30	0.25222929E 06
TOLUENE	10.442	%	40	0.84285395E 05
ALCOHOL	17.115	%	59	0.1581456E 06
WATER	39.989	%	72	0.3227616E 06
GLYCOL	3.672	%	78	0.2963900E 05

JOB COMPLETE

C.8

```
LIST   JOB        24        1        0  0.00000000E 00        0
JOB NO. =  24
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    5
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.50000023E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.29999992E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =    0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=     29
CNTRL DATA 1ST FILE REC. NO. =  39

                        TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION    FACTOR           AREA          COMPONENT
 TIME BAND     TIME BAND     FOR STANDARD                  CALC. CODE          NAME
0.500000E 00 0.116666E 01 0.000000E 00 0.998999E 01                    1    BENZENE
0.116666E 01 0.156666E 01 0.000000E 00 0.998999E 01                    1    TOLUENE
0.156666E 01 0.213333E 01 0.000000E 00 0.998999E 01                    1    ALCOHOL
0.213333E 01 0.250000E 01 0.000000E 00 0.998999E 01                    1    WATER
0.250000E 01 0.300000E 01 0.000000E 00 0.998999E 01                    4    GLYCOL

  COMPONENT      SEQ.  ADD AREA   FACTOR FROM   NINE =    POS. NO.-     UNITS
    NAME         NO. TO SEQ. NO.   SEQ. NO.    RF. PK.   CALC.CONC.     USED
                                                          BY DIFF.
BENZENE            1        0           0          9          0
TOLUENE            2        0           0          0          0
ALCOHOL            3        0           0          0          0
WATER              4        0           0          0          0
GLYCOL             5        2           0          0          0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     50

REF. PK. TIME TOL. VAR. NO. =     51

REL. TIME JOB FIN. VAR. NO. =     52

                        TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1                53                54
      2                55                56
      3                57                58
      4                59                60
      5                61                62
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  3.1660

THIS FUNCTION COMPLETE

ENDJ               0        0        0  0.00000000E 00        0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
 8   9
( 1)(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
     0   2   4   0   1


 * GAS CHROMATOGRAPH MONITORING *          ANALYST NO /   0
 0000  20/ 0/68                            JOB NO /     24
                                           INST / NO.    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
BENZENE	24.254	%	20	0.2172116E 06
TOLUENE	22.291	%	38	0.7777748E 05
ALCOHOL	14.566	%	58	0.1504504E 06
WATER	35.495	%	71	0.3178823E 06
GLYCOL	3.392	%	77	0.3038100E 05

JOB COMPLETE

C.9

```
LIST   JOB        55       1       0  0.00000000E 00       0
JOB NO. =  55
CHROM. NO. =    1
ANAL. CALC. NO. =  1
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =     3
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000000E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999996E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =    0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF C PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=      34
CNTRL DATA 1ST FILE REC. NO. =  42


                         TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION    FACTOR         AREA        COMPONENT
 TIME BAND     TIME BAND     FOR STANDARD                 CALC. CODE       NAME
0.700000E 00 0.119999E 01 0.999999E 02 0.000000E 00                1    PROPANE
0.115000E 01 0.155000E 01 0.999999E 02 0.000000E 00                1    BUTANE
0.150000E 01 0.225000E 01 0.999999E 02 0.000000E 00                1    PENTANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.   SEQ. NO.  RF. PK.  CALC.CONC.    USED
                                                       BY DIFF.
PROPANE            1       0          0         9         0
BUTANE             2       0          0         0         0
PENTANE            3       0          0         0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =      10

REF. PK. TIME TOL. VAR. NO. =     11

REL. TIME JOB FIN. VAR. NO. =     12

                         TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1               13                14
      2               15                16
      3               17                18
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ               0       0       0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
(  1)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   5   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *        ANALYST NO.    0
 0000  20/ 0/68                          JOB NO.     55
                                         INST. NO.    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	0.17099E-02	FACTOR	20	0.5848000E 05
BUTANE	0.14600E-02	FACTOR	28	0.6849130E 05
PENTANE	0.80601E-03	FACTOR	34	0.1240668E 06

JOB COMPLETE

Standardization Run Job Definition Cards

```
DFIN    VAR                                                13
   5001REF. TIME          3000+01
   5101TOLERANCE          5000-01
   5201FIN. TIME          9500+01
   5301MIN. TME 1         1500+01
   5401MAX TME 1          3500+01
   5501MIN TME 2          3500+01
   5601MAX TME 2          4700+01
   5701MIN TME 3          4700+01
   5801MAX TME 3          6400+01
   5901MIN TME 4          6400+01
   6001MAX TME 4          7500+01
   6101MIN TME 5          7500+01
   6201MAX TME 5          9000+01


DFIN    JOB
055  3  0 1                                   01    10    11    12 4
 PROPANE            1 1          1000+029 (             13    14
 BUTANE             2 1          1000+02  (             15    16
 PENTANE            3 1          1000+02  (             17    18
ENDJ
```

C.10

```
LIST    JOB         56        1        0  0.00000000E 00        0
JOB NO. =  56
CHROM. NO. =   1
ANAL. CALC. NO. =  2
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    3
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999996E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =    0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=     33
CNTRL DATA 1ST FILE REC. NO. =  35

                          TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND   TIME BAND    FOR STANDARD                 CALC. CODE       NAME
0.700000E 00 0.119999E 01 0.999999E 02 0.000000E 00             1   PROPANE
0.115000E 01 0.155000E 01 0.999999E 02 0.000000E 00             1   BUTANE
0.150000E 01 0.225000E 01 0.999999E 02 0.000000E 00             1   PENTANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.   USED
                                                        BY DIFF.
PROPANE           1       0           0         9        0
BUTANE            2       0           0         0        0
PENTANE           3       0           0         0        0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     20

REF. PK. TIME TOL. VAR. NO. =     21

REL. TIME JOB FIN. VAR. NO. =     22

                          TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1              13                 14
      2              15                 16
      3              17                 18
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ               0        0        0  0.00000000E 00        0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8  9
( 1)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   6   0   1


 * GAS CHROMATOGRAPH MONITORING *          ANALYST NO.   0
 0000  20/ 0/68                            JOB NO.     56
                                           INST. NO.   1

 PERCENT OF TOTAL AREA UNKNOWN =      0.00

 COMPONENT       RESULTS   UNITS     ELUTION TIME     PEAK AREA

PROPANE       0.10000E 01 FACTOR               21    0.5704633E 05

BUTANE        0.82152E 00 FACTOR               30    0.6943955E 05

PENTANE       0.44942E 00 FACTOR               36    0.1269512E 06

                      JOB COMPLETE
```

C.11

```
LIST   JOB        57       1       0  0.00000000E 00       0
JOB NO. =  57
CHROM. NO. =    1
ANAL. CALC. NO. =  3
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    3
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.20599997E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =    0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=     40
CNTRL DATA 1ST FILE REC. NO. =  48


                        TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION    FACTOR          AREA         COMPONENT
 TIME BAND     TIME BAND    FOR STANDARD                  CALC. CODE         NAME
0.700000E 00 0.119999E 01 0.000000E 00 0.100000E-04              1    PROPANE
0.115000E 01 0.155000E 01 0.000000E 00 0.100000E-04              1    BUTANE
0.150000E 01 0.225000E 01 0.000000E 00 0.100000E-04              1    PENTANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME        NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.    USED
                                                       BY DIFF.
PROPANE           1      0           0         9         0
BUTANE            2      0           0         0         0
PENTANE           3      0           0         0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     20

REF. PK. TIME TOL. VAR. NO. =     21

REL. TIME JOB FIN. VAR. NO. =     22

                        TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1               23                24
      2               25                26
      3               27                28
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( ) )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   7   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *        ANALYST NO.   0
 0000  20/ 0/68                          JOB NO.      57
                                         INST / NO.    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	0.600	%	21	0.6007008E 05
BUTANE	0.723	%	29	0.7230574E 05
PENTANE	1.293	%	35	0.1293094E 06

JOB COMPLETE

C.12

```
LIST   JOB       58       1       0  0.00000000E 00       0
JOB NO. =  58
CHROM. NO. =   1
ANAL. CALC. NO. =  5
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    3
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.20199999E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=     43
CNTRL DATA 1ST FILE REC. NO. =  51


                      TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION    FACTOR           AREA         COMPONENT
 TIME BAND     TIME BAND     FOR STANDARD                  CALC. CODE        NAME
0.700000E 00 0.119999E 01 0.000000E 00 0.100000E 01                 1    PROPANE
0.115000E 01 0.155000E 01 0.000000E 00 0.100000E 01                 1    BUTANE
0.150000E 01 0.225000E 01 0.000000E 00 0.100000E 01                 1    PENTANE

  COMPONENT     SEQ.  ADD AREA   FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.    USED
                                                        BY DIFF.
PROPANE           1      0           0          9          0
BUTANE            2      0           0          0          0
PENTANE           3      0           0          0          0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     20

REF. PK. TIME TOL. VAR. NO. =     21

REL. TIME JOB FIN. VAR. NO. =     22

                      TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME   REL. HIGH TIME
      1                23                24
      2                25                26
      3                27                28
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( 4)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
     0   5   8   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *        ANALYST NO.    0
 0000  20/ 0/68                          JOB NO /    58
                                         INST / NO /   1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	23.123	%	20	0.58850706E 05
BUTANE	27.161	%	28	0.69128617E 05
PENTANE	49.715	%	34	0.12655317E 06

JOB COMPLETE

C.13

```
LIST   JOB       59      1      0  0.00000000E 00      0
JOB NO. =  59
CHROM. NO. =   1
ANAL. CALC. NO. =  6
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =   3
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.99999953E 02
REF. PK. FACT. =0.10000002E 01
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19699999E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=    49
CNTRL DATA 1ST FILE REC. NO. =  56


                        TIME BAND DATA

 LOW LIMIT    HI LIMIT     CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND     FOR STANDARD                 CALC. CODE       NAME
0.700000E 00 0.119999E 01 0.999999E 02 0.999999E 00               1   PROPANE
0.115000E 01 0.155000E 01 0.999999E 02 0.841829E 00               1   BUTANE
0.150000E 01 0.225000E 01 0.999999E 02 0.456708E 00               1   PENTANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME        NO. TO SEQ. NO.   SEQ. NO.   RF. PK.  CALC.CONC.   USED
                                                       BY DIFF.
PROPANE           1      0           0          9         0
BUTANE            2      0           0          0         0
PENTANE           3      0           0          0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =    20

REF. PK. TIME TOL. VAR. NO. =    21

REL. TIME JOB FIN. VAR. NO. =    22

                         TIME BAND VARIABLE NO. DATA

TIME BAND NO.   REL. LOW TIME   REL. HIGH TIME
      1               23               24
      2               25               26
      3               27               28
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ               0      0      0  0.00000000E 00      0
END OF JOB
```

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2 CYCLE TIME
ONLY -- 5 OMIT
5
(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
  8   9
( ) )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
      0   5   9   0   1
```

```
* GAS CHROMATOGRAPH MONITORING *        ANALYST NO.   0
0000  20/ 0/68                          JOB NO.    50
                                        INST. NO.   1

PERCENT OF TOTAL AREA UNKNOWN =      0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	0.99999E 00	FACTOR	10	0.1411626E 06
BUTANE	0.64162E 00	FACTOR	27	0.1677005E 06
PENTANE	0.45670E 00	FACTOR	35	0.5001500E 06

JOB COMPLETE

C.14

```
LIST   JOB        60       1       0  0.00000000E 00       0
JOB NO. =  60
CHROM. NO. =   1
ANAL. CALC. NO. =  7
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    3
NORM. CONST. =0.00000000E 00
CONC. OF PK. =0.99999953E 02
REF. PK. FACT. =0.10000002E 01
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999996E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=     49
CNTRL DATA 1ST FILE REC. NO. =  53


                      TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND    FOR STANDARD                 CALC. CODE       NAME
0.700000E 00 0.119999E 01 0.999999E 02 0.000000E 00              1   PROPANE
0.115000E 01 0.155000E 01 0.999999E 02 0.000000E 00              1   BUTANE
0.150000E 01 0.225000E 01 0.999999E 02 0.000000E 00              1   PENTANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =  POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.  RF. PK.  CALC.CONC.   USED
                                                      BY DIFF.
PROPANE           1      0           0        9         0
BUTANE            2      0           0        0         0
PENTANE           3      0           0        0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     20

REF. PK. TIME TOL. VAR. NO. =     21

REL. TIME JOB FIN. VAR. NO. =     22

                      TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1                23                24
      2                25                26
      3                27                28
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  2.5000

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2 CYCLE TIME
ONLY -- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   0
( )=)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
    0   6   0   0   1
```

```
* GAS CHROMATOGRAPH MONITORING *        ANALYST NO.   0
0000  20/ 0/68                          JOB NO.     60
                                        INST. NO.    1

PERCENT OF TOTAL AREA UNKNOWN =     0.00
```

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	99.999		20	0.1446504E 06
BUTANE	100.158		29	0.1725146E 06
PENTANE	108.565		35	0.5125632E 06

JOB COMPLETE

C.15

```
LIST   JOB       61       1       0  0.00000000E 00       0
JOB NO. =  61
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    1
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.10000005E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.19999996E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =   0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =   4
TIME BAND DATA 1ST FILE REC. NO.=     49
CNTRL DATA 1ST FILE REC. NO. =  57


                        TIME BAND DATA

 LOW LIMIT    HI LIMIT    CONCENTRATION   FACTOR          AREA        COMPONENT
 TIME BAND    TIME BAND    FOR STANDARD                 CALC. CODE      NAME
0.700000E 00 0.119999E 01 0.000000E 00 0.150000E-02               1   PROPANE

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =  POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.   RF. PK.  CALC.CONC.   USED
                                                       BY DIFF.
PROPANE            1       0           0         9         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =     20

REF. PK. TIME TOL. VAR. NO. =     21

REL. TIME JOB FIN. VAR. NO. =     29

                        TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1               23                  24
NO CNTRL. ACT. SPECIF.
ABS. TIME PRG. BEGINS LOOKING FOR PKS.= 10.0000SEC.
REL. TIME JOB FINISH =  1.2998

THIS FUNCTION COMPLETE

ENDJ                0       0       0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
  8    9                                          63    2    1
(  #)(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
       0    6    1    0    1
```

63

	0163	LACO			
0:01		INPUT	=	+ 73.9	DEG.F.
0:01		INPUT	=	+ 73.9	DEG.F.
0:01		INPUT	=	+ 73.9	DEG.F.
0:02		INPUT	=	+202.5	DEG.F.
0:02		INPUT	=	+202.5	DEG.F.
0:02		INPUT	=	+202.5	DEG.F.
0:02		INPUT	=	+202.5	DEG.F.
0:02		INPUT	=	+202.5	DEG.F.

C.16

```
LIST   JOB        33       1       0  0.00000000E 00       0
JOB NO. =  33
CHROM. NO. =   1
ANAL. CALC. NO. =  4
NO. OF TIME BANDS + SPACE FOR UNK. PKS. =    2
NORM. CONST. =0.99999953E 02
CONC. OF PK. =0.00000000E 00
REF. PK. FACT. =0.00000000E 00
FRACTL. TOL. OF REF. PK. =0.50000023E 00
EXPECT. ABS. TIME OF REF. PK.(SECS) =0.94999969E 02
CALC. NO. USING OTHER ANAL. RSLTS. FOR FNL. REPT. (= 0 IF NONE REQD.) =    0
LOGICAL UNIT ANAL. REPT. PRT. ON (IF 0 PRT. ON UNIT NEAREST CHROM.) =    4
TIME BAND DATA 1ST FILE REC. NO.=     56
CNTRL DATA 1ST FILE REC. NO. =  66


                        TIME BAND DATA

 LOW LIMIT     HI LIMIT     CONCENTRATION   FACTOR          AREA         COMPONENT
 TIME BAND    TIME BAND     FOR STANDARD                  CALC. CODE        NAME
0.947368E 00 0.178947E 01 0.000000E 00 0.100000E 01               1   METH ALCOHOL
0.189473E 01 0.294736E 01 0.000000E 00 0.100000E 01               1   ETH ALCOHOL

  COMPONENT     SEQ.  ADD AREA  FACTOR FROM  NINE =   POS. NO.-    UNITS
    NAME         NO. TO SEQ. NO.  SEQ. NO.  RF. PK.  CALC.CONC.    USED
                                                       BY DIFF.
METH ALCOHOL      1       0           0         9         0
ETH ALCOHOL       2       0           0         0         0
VAR. NO. INFO.

REF. PK. EXPECT. TIME VAR. NO. =   203

REF. PK. TIME TOL. VAR. NO. =   204

REL. TIME JOB FIN. VAR. NO. =   205

                        TIME BAND VARIABLE NO. DATA

TIME BAND NO.    REL. LOW TIME    REL. HIGH TIME
      1               206              207
      2               208              209
1=REL. TM.    1=CHANG.     PARA.        NEW     1=ECO ON   ECO    ABS. OR REL.       TIME
0=ABS. TM.      PARA.       NO.        VALUE    0=ECO OFF  NO.     TIME TO DO      VAR. NO.
               0=ECO
    0             1           1           0       ----       --     0.148000E 02         210
    0             1           1           1       ----       --     0.848000E 02         211
REL. TIME FOR JOB FIN. =0.343100E 01

THIS FUNCTION COMPLETE

ENDJ              0       0       0  0.00000000E 00       0
END OF JOB
```

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
  8   9
(  4)(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )(   )
       0   3   3   0   1
```

```
 * GAS CHROMATOGRAPH MONITORING *          ANALYST NO /    0
 0000  20/ 0/68                            JOB NO /      35
                                           INST / NO /    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00

 COMPONENT         RESULTS   UNITS     ELUTION TIME      PEAK AREA

 METH ALCOHOL       52.122   %                  107    0.6661055E 06

 ETH ALCOHOL        47.877   %                  218    0.6116454E 06

                     JOB COMPLETE
```

C.17 Off-line Processing Technique

```
IF DATA TO BE ENTERED VIA CARDS ENTER 1 // ALREADY IN      FILE ENTER
1
PRESENT SCAN RATE = 5.00PTS/SEC ---ENTER NEW RATE       FORMAT(F5.2)
005
05.00



 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8   9
( 9 )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
      0   5   0   0   1



 * GAS CHROMATOGRAPH MONITORING *        ANALYST NO /    0
 0000  20/ 0/68                          JOB NO /      50
                                         INST / NO /    1

 PERCENT OF TOTAL AREA UNKNOWN =     0.00

 COMPONENT          RESULTS   UNITS      ELUTION TIME      PEAK AREA

 PROPANE             23.130   %                    20    0.1020425E 06

 BUTANE              27.226   %                    28    0.1201115E 06

 PENTANE             49.643   %                    34    0.2190100E 06

                  JOB COMPLETE
```

C.18 Repeated Analysis and 'Queuing Several Jobs' Features

```
 ENTER 1 FOR ANALYST OPERATING PROCEDURES-- 2CYCLE TIME
ONLY-- 3 OMIT
3
(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
  8    0                                  1    2   50   51   52
( 1)(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )(  )
      0    5    0    0    1



 * GAS CHROMATOGRAPH MONITORING *          ANALYST NO /    0
 0000  20/ 0/68                            JOB NO /      50
                                           INST / NO /    1

 PERCENT OF TOTAL AREA UNKNOWN =      0.00

 COMPONENT           RESULTS   UNITS      ELUTION TIME      PEAK AREA

 PROPANE              22.952   %                    19    0.5803520E 05

 BUTANE               26.887   %                    27    0.6900614E 05

 PENTANE              50.160   %                    33    0.1288591E 06

                       JOB COMPLETE



 * GAS CHROMATOGRAPH MONITORING *          ANALYST NO.     0
 0000  20/ 0/68                            JOB NO.       51
                                           INST / NO ,    1

 PERCENT OF TOTAL AREA UNKNOWN =      0.00

 COMPONENT           RESULTS   UNITS      ELUTION TIME      PEAK AREA

 PROPANE              23.646   %                    19    0.6156318E 05

 BUTANE               26.644   %                    27    0.6935836E 05

 PENTANE              49.709   %                    33    0.1294165E 06

                       JOB COMPLETE
```

* GAS CHROMATOGRAPH MONITORING *
0000 20/ 0/68

ANALYST NO / 0
JOB NO / 52
INST / NO / 1

PERCENT OF TOTAL AREA UNKNOWN = 0.00

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	27.237	%	19	0.6026763E 05
BUTANE	13.362	%	27	0.2956600E 05
PENTANE	59.400	%	33	0.1314351E 06

JOB COMPLETE

* GAS CHROMATOGRAPH MONITORING *
0000 20/ 0/68

ANALYST NO / 0
JOB NO / 50
INST. NO / 1

PERCENT OF TOTAL AREA UNKNOWN = 0.00

COMPONENT	RESULTS	UNITS	ELUTION TIME	PEAK AREA
PROPANE	23.145	%	20	0.5955298E 05
BUTANE	27.319	%	29	0.7029164E 05
PENTANE	49.534	%	35	0.1274513E 06

JOB COMPLETE

APPENDIX D

LITERATURE REVIEW

This appendix is a review of the literature relating to the subject of on-line computer chromatograph systems. The University of Alberta GC system is a multipurpose application, enabling GC monitoring, direct digital control and utilization of both these tasks to allow process control loops with chromatographs as the detecting elements. However, all the referenced material in this review describes the use of dedicated computer chromatograph systems to monitor and control laboratory and process chromatographs.

From the papers reviewed, three (25, 21, 13) are considered excellent material for workers in the field of on-line computer chromatograph systems. McCullough (25), the author of the IBM 1800 GC program describes his work in a clear manner. Frazer (21), gives one other excellent review in some detail of the many commercial and non-commercial computerized systems for laboratory automation. He discusses the various methods of automation such as data processing on or off-line with or without chromatograph control and presents the relative merits of small computers and large time shared machines for laboratory automation. Applications using three different computers, Varian 620i, DEC PDP-7 and IBM 1800, are described in this paper. This article does not give any indication of the successes of the described installations.

The following papers report the status of some of the present day commercial applications. Briggs (13, 24) and Chemical Engineering Progress (22), report that the IBM 1800 computer installation at Monsanto's plant has resulted in a considerable improvement in precision in many routine analyses. This installation, which uses the IBM GC program to

monitor 20 gas chromatographs simultaneously, is similar to the one at the University of Alberta in the Chemical and Petroleum Engineering department except for minor hardware differences. A typical example showed a change in standard deviation from 0.2 to 0.02 percent after the complete computer installation. The time required to generate a final report has been significantly reduced. This is particularly true of high speed capillary column chromatography which may resolve a hundred or more peaks. Some chromatograms, which previously required two hours to measure and calculate the results, are now typed out in three minutes. Briggs also reports that analysts have been relieved of the chore of monitoring a recorder to determine cut points and attenuation factors and reruns required because of missed attenuations have been eliminated.

Mears (16), reports that a small digital computer, dedicated solely to the control and analysis of data from process chromatographs has been operating on process streams at the Torrance, California refinery of Mobil Oil Corporation since April 1967. The final system is operating successfully on 8 gas chromatographs and a total of 27 process streams. The experience to date on the system has verified its feasibility in 1) controlling multiple process chromatographs and accurately reducing the data for presentation in an easy to understand format, and 2) proving a system of this type is maintainable by refinery maintenance personnel.

Dietz (17), reports that a high accuracy, computer chromatograph system, consisting of a small general-purpose dedicated computer and seven single stream chromatographs, is being used for analyzing key components produced from a light hydrocarbon and crude oil feed stock. The system has proved to be a valuable aid to more accurate process distillation operation. The reported accuracy of the computer chromatograph system is

stated to be ± 0.1% of the value of the variable plus 0.05% for overall system error. A table in this article shows that data taken over a two week period, reporting two results per day, has an average deviation of 0.09 for propane from the true value of 8.28%. No comparison is made between a conventional type chromatograph system and the computer chromatograph system in this reference.

Harris, Hickerson and Morgan (18), report that a real-time monitoring computer has been installed in the Humble Oil and Refining Company's Baytown laboratory. This system monitors 11 gas chromatographs, one mass spectrometer and one nitrogen analyzer. A table of seven repeated analysis runs is shown for a six component chromatograph analysis and a maximum of 1.2% standard deviation was reported. The authors feel that is is advantageous to use a relatively large computer, core size 16K, because of the versatility that is offered. No comparison is reported between the original system and the computer chromatograph system.

Felton, Hancock and Knupp Jr. (20), and Chemical and Engineering News (19), report that Du Pont's Jackson laboratory have installed an EAI computer chromatograph system capable of processing data from 40 gas chromatographs simultaneously. This system has eliminated a large calculation backlog and created major cost savings. The heart of the hardware for this system is a PDP-8 high speed digital computer, which has an 8000 work core memory. Du Pont chemists point out that work of the analytical chromatographic group requires 25% fewer technicians than before, and although output has increased from about 40 chromatograms to 60 chromatograms per eight hour shift the calculation backlog has disappeared.

The following references discuss the hardware and/or software of computer chromatograph systems. Fraade (23), presents a general article

indicating the functions that the on-line computer can perform which is now presently carried out by hardware in the process chromatographs. Mears (26), describes the problems of connecting process gas chromatographs to a computer without an interrupt feature. He then discusses how the interrupt system simplifies the hardware necessary in a process analyzer to provide adequate communication with the computer.

Although process control was not a part of this thesis, the author would like to reference a paper for future computer process control chromatograph researchers. Ogle (27), presents a good article which describes his experimentation with on-line computer chromatograph control systems. He describes the problems involved and the advantages of the system from his experimental work.

Since control systems utilizing chromatographs are sampled data in nature, two good texts (28, 29) for sampled data analyses are referenced. There are numerous references to gas chromatography in the literature but the author has made no attempt to review them for this thesis.

B29905